**AFRL-ML-WP-TR-1999-4040** 

TESTING OF AIRCRAFT RUNWAY ICE CONTROL PRODUCTS

**MATERIALS COMPATIBILITY** 



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# EXECUTIVE SUMMARY

New commercial formulations of runway ice control products have recently made their way onto military and commercial aircraft runways. These new ice control products are purchased to AMS/SAE (Aerospace Materials Specifications/Society of Automotive Engineers) 1431 and 1435. These products have not been tested for compatibility with a number of aircraft materials, many of which are common to both commercial and military aircraft. In addition, there are some materials that are unique to military aircraft; infrared windows, for example, for which no compatibility testing had been done. To fill this knowledge gap AFRL/MLSA undertook a study to evaluate the compatibility of these products with aerospace materials. The compatibility testing performed on these products exceeds the AMS/SAE specification requirements. This report documents AFRL/MLSA's effort to identify possible problems for both commercial and military aircraft with new deicer/anti-icer materials not evident with the previously used ice control materials.

### **ACKNOWLEDGMENTS**

We would like to thank the following individuals for their assistance in this effort:

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Eddie White--AFRL/MLSA

Mary Wyderski--ASC/EME

# Testing of Aircraft Runway Ice Control Products (Materials Compatibility)

30 October 1998

Evaluation Report (4349IHRD/S410)

Report No. AFRL/MLSA 98-137

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### **Testing of Aircraft Runway Ice Control Products**

### **PURPOSE**

Determine if the new runway deicers/anti-icers (potassium acetate, sodium acetate and sodium formate) adversely affect the material/mechanical properties of various aircraft materials when subjected to laboratory tests.

### **BACKGROUND**

The Air Force has been, and is now, procuring new environmentally compliant runway deicer/anti-icer chemicals to the requirements of AMS 1431 and 1435. The specifications do not require the effect of these chemicals on aircraft structural and nonstructural materials. The specifications are designed mainly for commercial aircraft use.

A number of aircraft materials, common to commercial and military aircraft, in addition to to some unique to military aircraft (e.g., infrared windows), have not been tested for compatibility with the new ice control chemical products

### FACTUAL DATA

Five runway deicer/anti-icer materials were used in this evaluation (table 1). Systems Program Office (SPO) engineers identified aircraft materials thought to be potentially affected by these materials. The aircraft materials were categorized and analyzed for similarities to AMS requirements by AFRL/ MLS engineers. Materials to be tested (table 2) had to be common to more than one weapons system. Those that were system unique are to be tested in the future by the SPO.

Testing was to be done at the AFRL Materials and Manufacturing Directorate, Raytheon Technical Services Co., Indianapolis, Indiana and at Hill Air Force Base, Utah (brakes only). Individual reports for each type of material tested are attached to this summary as appendices.

### **DISCUSSION(S)**

CAUTION: Testing results in this report are in some cases "worst case scenarios." In actual applications on aircraft there are usually protective coatings on these materials, or they are not exposed to the external environment. Therefore, testing on bare or unprotected material, as in these tests, represents a "worst case scenario."

### CONCLUSION(S)

The following conclusions are brief overviews. Please see individual appendices for detailed results.

### Metals Testing

The laboratory testing provided a ranking of the reactivity of deicing chemicals towards aerospace structural materials. Magnesium alloy exposure to AMS ice control materials is a concern.

Potassium acetate and sodium formate were particularly corrosive to cast magnesium alloy AZ91E when compared to the control chemicals ethylene-glycol-UREA and UREA. The accelerated corrosion can be managed, if facilities are available during cold weather to inspect and clean exposed magnesium components. Removal of deicing chemicals should be accomplished in accordance with Technical Order (T.O.) 1-1-691 (General Corrosion Requirements) as soon as possible after exposure. Regular inspection and maintenance of coating system integrity of the magnesium components needs to occur throughout the year. System specific T.O. -23s (Corrosion Technical Orders) may need to address cleaning/removal procedures for vulnerable magnesium components over and above the general guidance in T.O. 1-1-691.

Society of Automotive Engineers (SAE) Committee G-12 should consider the use of alternate immersion per ASTM F482 for metals compatibility testing. In addition, this committee should choose the most widely used magnesium alloy for compatibility testing. AZ31B is rarely used in service but is targeted in the AMS 1431 and AMS 1435 specifications. Assistance in determining the alloys most frequently used should be sought from the Magnesium Trade Association.

### Composites Testing

The S2/AFR700B glass fiber composite saw an approximate 10 percent reduction in ultimate shear strength and a 5 percent reduction in shear strength at 5 percent strain when exposed to the potassium acetate and sodium acetate in laboratory conditions. In open-hole compression strength tests, the S2/AFR700B material saw a 30 percent reduction in the potassium acetate, 22 percent in the sodium formate and 18 percent in the sodium acetate based ice control chemicals. System program managers should review the results in evaluation report MLS-98-126, dated 27 July 1998, (appendix 2) of this report.

### Infrared Window Testing

Sodium formate should not be used as a deicer/anti-icer fluid for Air Force systems that use exposed Ge, Poly-Si, or ZnS IR windows. The deicer/anti-icer fluid will stain the window, reduce its transmission and thus the performance of the system. If sodium formate must be used, it is recommended that an additional cleaning study be performed to determine whether or not a solvent exists that will remove the sodium formate stains from these window materials.

Managers, owners and operators of systems containing protective covers or other approaches to protect sensitive optical materials from the environment must ensure they will prevent exposure to environmentally acceptable ice control materials.

Advanced sensor windows will include complex coatings to meet optical and electromagnetic interference requirements. Exposure to environmental hazards such as raindrop impacts, hail, dust or runway debris will damage the coatings and expose the constituent materials to chemical attack by deicing fluids and cleaning solvents. Any cleaning or deicing compounds considered for use on military aircraft that will come in contact with airborne sensor windows should be tested for compatibility with the window materials and coating systems.

System managers relying on infrared or advanced sensor windows must consider the data presented in this report to evaluate specific weapon system issues and determine whether additional testing is required.

### Elastomers and Sealants

A serious loss (90-100 percent) in peel strength was evident with the exposure of PR-1750 B2 sealant to all the deicers/anti-icers. Additional testing is being performed to identify the basis for this incompatibility. This sealant should not be used in any area where it could be exposed to deicers/anti-icers such as environmental sealing in wheel wells or aerodynamic sealing on the outer mold line. PRC 1750B-2, i.e., AMS 3276 or MIL-S-83430, is still acceptable for sealing integral fuel tanks where there is no exposure to these ice control chemicals. Acceptable substitutes for PRC 1750, which are resistant to the ice control chemicals per appendix 4, are products qualified to AMS-S-8802, and should be reviewed by system program managers for at least the B-1, F-16 and F-15. For applications where the service temperatures exceed 225°F sealant products qualified to AMS 3277 are recommended.

The four percent decrease in cohesive strength for PS-870B2 sealant material, when exposed to sodium acetate based chemicals, is of little concern. No significant changes in properties of either nitrile or neoprene bladder materials were observed.

### **Electronics**

The wire insulation materials tested showed no detrimental effects from the deicers/anti-icers. Following immersion, all wires passed the bend and voltages withstand test. All deicer/anti-icer materials (except UREA) failed the wet arc track propagation resistance test in varying degrees. Most Air Force aircraft contain a wire insulation construction (MIL-W-81381) that is susceptible to arc tracking. The conductivity of the AMS deicer/anti-icer materials is in the range of a three- percent salt solution. Materials with this conductivity level could cause malfunctions in any electrical equipment. All electrical systems may thus be susceptible to failure resulting from exposure to these ice control materials.

Replacement deicers/anti-icers should have a maximum electrical conductivity value. An

acceptable conductivity value will need to be determined through additional testing. At this time the UREA value in the range of 600E-6 S/cm should be the goal. Procedures should be developed to minimize exposure of electronic systems, wiring and connectors. A field evaluation should be conducted on those military systems, which have been operated from an airfield treated with these AMS materials to determine if a problem currently exists. Controlled deicer/anti-icer application and training on its effect on electrical systems may minimize electrical failures.

### Carbon-Carbon Brakes

Test results from three brake manufacturers' materials confirmed that the new deicers/anti-icers have a significant detrimental effect on carbon-carbon materials when exposed to typical 1300°F temperatures. In order of severity, the new deicers/anti-icers had the greatest effect followed by the glycol mix. All uncoated areas, which comprise the majority of the brake wear surfaces, lost hardness when exposed to the new deicers/anti-icers. Any loss of hardness is detrimental to the structural integrity of the carbon-carbon materials and will reduce wear performance capability. Those systems, including B-2, C-17, C-5, F-15, F-16 and F-22, using the carbon-carbon brake materials should re-evaluate their inspection intervals. Inspection intervals on the brake wear surfaces should be reduced to accommodate the reduction in wear performance during cold seasons when exposure to these deicers/anti-icers is a possibility. Long-term effects of carbon-carbon exposure to the new deicers/anti-icers were not evaluated.

### **RECOMMENDATION(S)**

Based on the above comments, users, whose aircraft are potentially exposed to these new ice control materials, should check their aircraft inventory for potential compatibility/corrosion problems.

Operators should be aware of those specific chemicals used on all runways they encounter and should assess any risks. Be advised that ice control chemical manufacturers do periodically change formulations and this will lead to future unknowns.

Maintainers may wish to consider periodic fresh water rinses of aircraft to preclude problems in certain aircraft.

This testing was not exhaustive. Cognizant engineering authority and system operators are responsible for testing those materials that are unique to their particular aerospace system.

Carbon-carbon brake materials appear to be the most affected components in the study. Reduced brake pad life can be expected.

Recommend individual users contact the authors of the appendices for specific questions and guidance.

**TABLES** 

### Table 1

Runway Deicer/Anti-icer Materials Used for All Tests

Propylene Glycol-UREA (Control-liquid) UREA (Control-solid) Liquid Potassium Acetate Solid Sodium Acetate Solid Sodium Formate

### Table 2

### Aircraft Materials Tested

### Metals

A286 steel
9Ni-4Co steel
PH 13-8 steel
4140 steel
AZ91E magnesium alloy
C99300 aluminum/bronze alloy
7075 bare aluminum

### Composites

AS4/3501-6 graphite/epoxy IM7/5250-4 graphite/ bismaleimide S2/AFR700 fiberglass

### Infra Red Windows

REP/DAR Coated Zinc Sulfide Boron Phosphide Coated Zinc Sulfide Polymer Bond with Zinc Sulfide and Polysilicon Clad Zinc Selenide

Poly-silicon Aluminum Oxynitride (ALON) Germanium

### Elastomers(Mil-R-6855)

Nitrile (Class 1) Neoprene (Class 2)

### **Sealants**

Corrosion inhibiting (PS-870 B-2) Fluorosilicone (QA-2817) Polysulfide (PR1422 B-2) Polysulfide (PR 1750 B-2) Polythioether (PR1826 B-1/2)

### Table 2 (contd)

### **Electronics**

Insulated wiring

Hybrid Polyimide Teflon® 22759

Printed Wiring Boards

Glass epoxy with no conformal coat Glass epoxy with a polyurethane conformal coat.

### Carbon/carbon Brake Pads

Carbon / carbon from three manufacturers with and without anti-oxidation treatment.

**APPENDICES** 

# APPENDIX 1 METALS

Compatibility of New Runway Deicers on Aircraft Structural Metals (Materials Evaluation)

11 August 1998

**Evaluation Report** (4349IHRD/APSS)

Report No. AFRL/MLSA 98-130

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### **EXECUTIVE SUMMARY**

This program was initiated at the request of ASC/EME and HQ AFCESA/CEOM to address concerns regarding material compatibility of newly acquired, environmentally friendly runway deicers. In response, a request was sent to all MAJCOMs to learn what structural materials will come in contact with, and possibly be compromised by, the deicers. It was agreed that materials unique to one weapon system would be tested for compatibility by that office and Wright Labs would test seven common alloys.

The new commercial deicers were tested directly against control deicers and the difference in performance was measured. Flat plates and stressed specimens were alternately immersed in the deicing chemical(s) for up to two weeks. A visual assessment of corrosion on the plates and cracking of the stress corrosion specimens was performed. Additional testing included electrochemical potentiodynamic polarization. Data obtained from the corrosion tests suggest there may be a compatibility problem with two of the deicers when in contact with magnesium alloy AZ91E. These deicers were significantly more reactive than the controls. Procedures to minimize corrosion of magnesium components when exposed to the deicers are recommended.

### **ACKNOWLEDGMENTS**

A note of appreciation is extended to Mr. Ken Chitwood of The University of Dayton Research Institute for his assistance with this investigation.

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### Compatibility of New Runway Deicers on Aircraft Structural Metals

### **PURPOSE**

Compare the corrosion resistance of representative aerospace metals when exposed to conventional and emerging runway deicer fluids.

### **BACKGROUND**

Environmentally compatible runway deicing chemicals have recently been incorporated at various commercial and military bases. These chemicals have been approved under commercial specifications AMS 1435 (liquid deicers) and AMS 1431 (solid deicers). Several system program offices (SPOs) requested that these deicers be evaluated for compatibility with aerospace materials used in weapon systems. Their concern is that the tests and alloys used by the commercial specifications were limited specifically to commercial aircraft applications and are not representative of military applications. Therefore, a test plan was developed by the Materials and Manufacturing Directorate of the Air Force Research Laboratory to address compatibility of the new commercial deicers with common Air Force aircraft materials using alternative test methods<sup>1</sup>. This report summarizes the compatibility evaluation effort.

### **Deicing Chemicals**

Five deicers were used in this study; two control chemicals, two new solid and one new liquid formulation. UCAR PM-5197 made by Union Carbide Corporation was the runway deicer most commonly used by the Air Force (procured under the now canceled specification, MIL-D-83411) and was used in this study as the liquid control. UCAR PM-5197 consists of approximately 50 percent ethylene glycol, 23 percent urea, 25 percent water and inhibitors. Unfortunately, ethylene glycol exerts a very high biochemical oxygen demand (BOD) on receiving waters depriving aquatic life of oxygen. Ethylene glycol is also toxic and a hazardous air pollutant, as defined under the Clean Air Act<sup>2</sup>. The solid control deicer used for baseline comparison was urea, consisting of 97-99 percent urea, manufactured by Agricultural Minerals Corporation. Urea is being phased out because of its high BOD and toxicity<sup>2</sup>.

Three new deicers formulated to alleviate these environmental concerns were evaluated in this study. They have been used in Europe and Canada, and their major constituents (though not specific formulations) are advocated for Air Force use <sup>2</sup>. The liquid deicer is Cryotech E36, manufactured by Cryotech Deicing Technology, and contains 50 percent potassium acetate and 50 percent water with less than 1 percent corrosion inhibitors. The two other deicers are in solid pellet form. Safeway SF is manufactured by Clariant Incorporated and consists of 98 percent minimum sodium formate with a corrosion inhibitor. Octagon RD1431 SA is manufactured by

<sup>&</sup>lt;sup>1</sup> Materials unique to a particular weapon system were left to that specific agency to conduct compatibility testing if deemed necessary.

<sup>&</sup>lt;sup>2</sup> Report referenced: Air Force Aircraft and Airfield Deicing/Anti-icing, Pro-Act document no. 17589.

Octagon Process Incorporated and contains less than 80 percent sodium acetate trihydrate in addition to sodium metasilicate and corrosion inhibitors.

Simulating typical airfield usage, the solid deicers were mixed with distilled water in the ratio of 15 grams of deicer per 100 ml of water to form an approximately 13 percent by weight solution. Liquid deicers were tested, as-received, with no dilution, again characteristic of airfield anti-icing usage.

### **FACTUAL DATA**

### Test Panels/Specimens

Seven alloys were selected for this study based on prevalence among weapon systems, reactivity in other corrosive mediums and material availability<sup>3</sup>. These included four steels (PH13-8, A286, 4140, and 9Ni-4Co), an aluminum alloy (7075-T6 no clad), an aluminum-bronze alloy (C99300) and a cast magnesium alloy (AZ91E-T6).

The chemical compositions of the alloys were verified by inductively coupled plasma-optical emission spectrometry. Tensile properties of the materials were verified to be within specification.

### Specimen Preparation

The alloys were machined to produce specimens for coupon testing and stress corrosion cracking (SCC) testing. Coupons measuring 5 x 5 cm and 2 x 2 cm squares were machined using electrostatic discharge machining (EDM) wire to avoid localized heating. The larger coupons were ground to a 32  $\mu$ in. rms finish, wiped clean with methyl ethyl Ketone (MEK), and weighed. The smaller coupons to be used for electrochemical measurements were polished with 120, 240, 320, 400 and 600-grit silicon-carbide paper to obtain a smooth surface of known surface area.

The SCC specimens were prepared in accordance with ASTM G-49, Standard Practice for Preparation and Use of Direct Tension Stress-Corrosion Test Specimens. Frames to hold the specimens under constant strain while exposed to the deicer were machined from 7075-T6 aluminum, which were then anodized per MIL-A-86285, Type I. Figure 1 shows the specimen and frame. Plate alloys were tested in the short transverse (ST) orientation.

<sup>&</sup>lt;sup>3</sup> Alloys tested for compatibility in AMS 1431 and AMS 1435 include 2024-T3 aluminum (bare and clad), 7075-T6 clad aluminum, AZ31B magnesium, Ti-6-4 titanium, and 1020 carbon steel.

### **Testing**

The AMS specifications for runway deicers cover a broad range of performance criteria. The purpose of this investigation was to augment the commercial specification requirement with additional test relevant to military applications. The tests used were alternate immersion, constant-strain stress corrosion cracking and electrochemistry. Each evaluation was performed in triplicate.

### Alternate Immersion

An alternate immersion test was used to cycle metals between wet and dry conditions to mimic similar cycling when runway deicers are sprayed onto the aircraft during take-off and landing. The test methodology was adopted from ASTM G-34, Standard Test Method for Exfoliation Corrosion Susceptibility in 2xxx and 7xxx Series Aluminum Alloys using deicer chemicals instead of sodium chloride solution as the test solution.

The 5 x 5 cm coupons were placed vertically in a tank of deicer solution. To eliminate cross contamination of corrosion product(s), the alloys were tested separately according to material class. Steel coupons were run separately from the aluminum, etc. The immersion cycle was 10 minutes wet and 50 minutes dry, continuous for two weeks. The exception was only one week for the magnesium alloy due to the amount of corrosion observed. At the end of the test the coupons were rinsed, dried and weighed. Before weighing the magnesium coupons, corrosion product was removed by immersion in a solution containing 20 percent chromium trioxide, 1 percent silver nitrate and 2 percent barium nitrate as described in ASTM G-1, Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens.

### Stress Corrosion Cracking

Stress corrosion cracking tests were performed in accordance with ASTM G-44, using the constant-strain method. Specimens were placed in frames and strained to 80 percent of yield stress using a stressing fixture and strain gauge. The 0.5 inch strain gauge, connected to a voltmeter, was placed in the gauge section of the specimen. As the fixture "squeezed" the frame, elongation of the frame pulled the specimen in tension until the desired strain was obtained. A wax was used to separate the two dissimilar metals. After two days of relaxation in the frame specimen, yield strengths reached a plateau of about 70 percent of yield stress. At this time the specimens mounted in their frames were immersed in the alternate immersion tank with their respective 5 x 5 cm coupons. Therefore, the cycling conditions were the same for the SCC specimens and the alternate immersion coupons. SCC specimens were removed at the time of fracture or after two weeks.

#### **Electrochemistry**

Potentiodynamic polarization scans were performed on three of the alloys that reacted with

the deicers in the alternate immersion test<sup>4</sup>. The polished 2 x 2 cm coupons of AZ91E, 4140 and 7075 were tested in each deicer formulation versus a saturated calomel reference electrode. Polarization scans were initiated 30 mV below the open circuit potential (OCP) and were continued by polarizing the sample anodically at 0.2 mV/sec. The experimental setup consisted of a flat cell, working, reference and counter electrodes, and a Solartron 1287 Electrochemical Interface.

### Results

The alternate immersion and electrochemical tests were useful in measuring the corrosivity of the new deicer formulations on these structural metals, while the SCC test was found to be less informative. Four of the alloys, A286, PH13-8, 9Ni-4Co and C99300 showed no signs of degradation in any of the test conditions.

### **Alternate Immersion**

Coupons from the alternate immersion test were visually examined for the amount and type of corrosion. As described in table 1, some form of degradation was found for seven conditions: AZ91E exposed to Cryotech E36, Urea, Safeway SF, and Octagon RD1431 SA; 4140 exposed to Urea and Safeway SF; and 7075 exposed to Urea. After removing the corrosion product(s) formed on the magnesium alloy, it was observed that those coupons exposed to Cryotech E36 and Safeway SF were heavily corroded, with weight losses of 3.1 percent and 4.5 percent, respectively. The predominant response of the AZ91E exposed to urea was observed in the form of pitting. The pits ran together along ridges left from the surface machining operation. The Octagon product caused only 5-7 pits per surface on AZ91E. Since the corrosion was in the form of pits for these two exposures, weight loss was negligible.

As expected from field reports, the 4140 alloy corroded significantly in the urea solid control deicer. Safeway SF caused only slight staining of the 4140.

Urea also degraded bare 7075-T6; staining its surface and exfoliating the machined edges.

### Stress Corrosion

The only stress corrosion cracking failure observed was on the AZ91E alloy exposed to Safeway SF. One specimen failed after approximately 164 hours. The other two failed during disassembly of the specimen and frame.

<sup>&</sup>lt;sup>4</sup> Polarization curves provide information on the corrosion behavior of a material in a time by monitoring the change in current density as a potential is incrementally increased between the material of interest (working electrode) and a reference electrode. The current response is a direct function of corrosion rate as the working electrode is oxidized, thereby releasing electrons. Additional information on the test method may be obtained from ASTM G-5, Standard Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements, and ASTM G-102, Standard Practice for Calculation of Corrosion Rates and Related Information from Electrochemical Measurements.

### Electrochemistry

Polarization curves of AZ91E, 7075 and 4140 in each of the deicers are shown in figures 2 through 4<sup>5</sup>. Predictions of corrosion behavior obtained from the polarization scans of AZ91E in the deicers correlate well with the corrosion observed on the alternately immersed coupons. For example, there was no passive region of AZ91E in Cryotech E36 or Safeway SF. The scans indicate active general corrosion, which was observed on the coupons from the immersion test. The slope of the curve for urea was greater, indicating less active corrosion compared to Cryotech E36 or Safeway SF, which is what was also observed on coupons. The curve of AZ91E in Octagon RD1431 SA showed passivation before breakdown at about -625 mV where pitting would be predicted to occur. A few pits were found on these coupons from the alternate immersion test. The UCAR deicer did not react with AZ91E. This inert behavior is predicted by the extremely large passive region of the polarization curve.

There was no passivation of 4140 in the urea deicer, and the corrosion current density was one to two orders of magnitude greater compared to the other deicers. This correlates with the heavy corrosion product observed on 4140 exposed to urea in the alternate immersion coupon test.

The curves of the 7075 alloy were very similar to one another. The large passive regions are indicative of the lack of corrosion observed in the coupon test, with the exception of the slight exfoliation in urea. As the exfoliation is believed to be a result of the alternate immersion exposure, the constant immersion exposure of the electrochemical experiment would not be expected to predict that result. Also, the exfoliation occurred on machined ends, whereas the polished surface planes were used for electrochemical testing.

### **DISCUSSION(S)**

Data obtained from the corrosion tests suggest there may be a compatibility problem with two of the deicers when in contact with AZ91E, since they were significantly more reactive than the controls. These deicers passed the current AMS specifications. A revision of the commercial specifications to account for the susceptibility of AZ91E, or other magnesium alloys used in military aircraft should be considered. Specification of the duration and/or nature of the immersion test should be determined by experiment when developing the revision to the commercial specification.

The electrochemical test proved to be a valuable accelerated technique for comparison of the alloy susceptibility to corrosion in the deicers. The results correlated well with coupon testing and the behavior of an alloy in a deicer can be obtained in less than a day. Although interpretation of the results may be more complicated, the use of a control deicer provides a good standard for comparison.

<sup>&</sup>lt;sup>5</sup> Alloy 4140 could not be tested in Octagon RD1431 SA due to a lack of available deicer material.

### **CONCLUSION(S)**

A series of corrosion tests were used to determine the compatibility of new solid and liquid runway commercial deicer formulations with common aerospace structural metals. Electrochemical measurements and alternate immersion tests were successful at distinguishing corrosion behavior of susceptible alloys amongst the deicer chemicals.

Four alloys (A286, PH13-8, 9Ni-4Co, C99300) were not effected by any of the deicers, and only magnesium alloy AZ91E reacted with the new deicers. Corrosion of AZ91E was more severe when exposed to Cryotech E36 and Safeway SF compared to the other deicers.

### **RECOMMENDATION(S)**

Consider precautionary measures if using Cryotech E36 or Safeway SF on aircraft containing exposed magnesium components. Ensure these parts have continuous protective coatings free of scratches and defects that would expose the bare metal. Cleaning/washing/fresh water rinsing of magnesium components after exposure to runway ice control materials is advised.

Revise commercial specification for runway deicers to include a test that can identify significant differences in deicer reactivity on magnesium alloys. Consider the use of an alternate immersion test that simulates wet and dry cycling of deicers on aircraft and electrochemical testing for accelerated evaluation of new deicer compatibility.

### **REVIEWED BY**

//signed//

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PUBLICATION REVIEW: This report has been reviewed and approved.

//signed//

MICHAEL F. HITCHCOCK, Branch Chief Materials Integrity Branch Systems Support Division Materials and Manufacturing Directorate

cc:

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# **FIGURES**

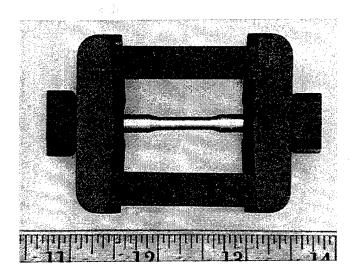


Figure 1. Constant strain/stress corrosion cracking specimen mounted in a chromic acid anodized aluminum frame.

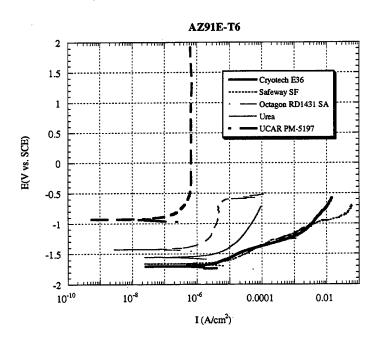


Figure 2. Potentiodynamic polarization curves of cast AZ91E-T6 in runway deicers. Urea was used as the control for the solid deicers, Safeway SF and Octagon RD1431 SA. Solid deicers were diluted with distilled water to 13 percent. UCAR was used as the liquid control.

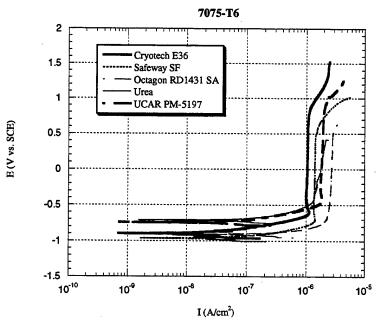


Figure 3. Potentiodynamic polarization curves of 7075-T6 in runway deicers. Urea was used as the control for the solid deicers, Safeway SF and Octagon RD1431 SA. Solid deicers were diluted with distilled water to 13 percent. UCAR was used as the liquid control.

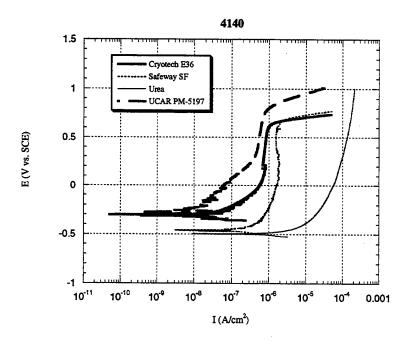


Figure 4. Potentiodynamic polarization curves of 4140 in runway deicers. Urea was used as the control for the solid deicer, Safeway SF. Solid deicers were diluted with distilled water to 13 percent. UCAR was used as the liquid control.

**TABLES** 

Table 1

Corrosion Observed On Alternate Immersion Coupons

	Cryotech E36 (liquid)	Safeway SF (solid)	Octagon RD1431SA (solid)	Urea (liquid control)	UCAR PM-5197 (solid control)
AZ91E	Shallow pits that ran together in regions. 3.1% weight loss.	Pits that ran together to look like general corrosion.	5-7 pits per side.	Small pits that gathered along ridges formed by machining.	No corrosion
		loss.			
7075	No corrosion	No corrosion	No corrosion	Staining on surface. Exfoliation shown here on cut edges.	No corrosion
4140	No corrosion	Slight staining in regions.	No corrosion	Major build up of corrosion product.	No corrosion
PH13-8	No corrosion	No corrosion	No corrosion	No corrosion	No corrosion
A286	No corrosion	No corrosion	No corrosion	No corrosion	No corrosion
9Ni-4Co	No corrosion	No corrosion	No corrosion	No corrosion	No corrosion
C99300	No corrosion	No corrosion	No corrosion	No corrosion	No corrosion

# APPENDIX 2 COMPOSITES

# Materials and Manufacturing Directorate System Support Division, AFRL/MLS Wright-Patterson Air Force Base, Ohio 45433-7718

#### **Evaluation Report**

# Exposure of Polymer Matrix Composites to Environmentally Friendly Runway Deicers

REPORT #: MLS-98-126

DATE: 27 JULY 1998

PROJECT #: 4349IHRD APSS

TYPE EVAL: Mechanical and Physical Property

**DISTRIBUTION STATEMENT A**: Approved for public release; distribution is unlimited.

#### 1.0 PURPOSE:

To evaluate the effect of environmentally friendly runway deicers on the mechanical and physical properties of composite materials.

#### 2.0 BACKGROUND:

The use of recently developed environmentally friendly runway deicers has resulted in a need to evaluate the effect of these materials on the mechanical properties of a wide range of aircraft materials. Several aircraft system program offices (SPOs) such as the B-2, C-17, and F16 are interested in learning the effect of recently developed, more environmentally friendly runway deicers on possible induced corrosion and changes in material mechanical properties. This report will cover the effort to examine the effect of absorbed runway deicer on the mechanical and physical properties of polymer matrix composites.

Deicers come in two physical forms, solid and liquid. The solid deicers are helpful for ice removal since they may melt through the ice then continue to melt along the ice runway surface which allows the ice to be removed with a snow plow. Liquid deicers are used for anti-icing after the snow or ice has been removed.

Two popular liquid runway deicers are UCAR PM-5197 and Cryotech E-36. UCAR PM-5197 is made by Union Carbide Corporation and consists of approximately 50% ethylene glycol, 23% urea, and 25% water with inhibitors. In the past, this material has been the most commonly used runway deicer and was evaluated as the liquid deicer control for this effort. E-36 was developed as a more environmentally friendly liquid runway deicer and is manufactured by Cryotech. E-36 contains 50% potassium acetate and 50% water with less than 1% corrosion inhibitors.

Three solid deicers used were Urea, Safeway SF, and Octagon RD1431 SA. To simulate the effect of the solid deicer mixing with snow or ice then being transferred to the aircraft, the solid deicers were mixed with distilled water in the ratio of 15 grams of deicer per 100 ml of water to form an approximate 13% by weight solution. The solid urea was manufactured by Agricultural Minerals Corporation and consisted of 97-99% urea. Urea was the control for all solid deicers. Safeway SF was manufactured by Clariant (Canada) Incorporated and consisted of 98% (minimum) sodium formate with a corrosion inhibitor. Octagon RD1431 SA was manufactured by Octagon Process Incorporated and contained less than 80% sodium acetate trihydrate. Testing was also performed on specimens that were vacuum oven dried and not exposed to deicer fluid for baseline data. Table 1 summarizes the runway deicers studied in this investigation.

Three, different composite materials were evaluated in this study. The materials included AS4 (Graphite) / 3501-6 (Epoxy), IM7 (Graphite) / 5250-4 (Bismaleimide), and S-2 (Glass) / AFR700B (Polyimide). The materials were fabricated into panels that were evaluated for quality prior to being machined into the appropriate test specimens.

The effect of the runway deicers on composite materials was evaluated in this study by performing both mechanical and physical testing. The mechanical testing included In-Plane Shear (IPS) strength and Open-Hole Compression (OHC) strength. The IPS testing was performed at both room temperature and elevated temperature while all OHC testing was performed at room temperature. Physical testing included Barcol hardness, glass transition temperature, cross-sectional analysis, sandwich corrosion, galvanic corrosion, and thermal oxidative stability. Table 2 depicts the material/test matrix and the number of specimens tested per deicer fluid.

# 3.0 TEST PANELS / SPECIMENS:

Panels were fabricated from three different composite materials (AS4/3501-6, IM7/5250-4, and S-2 Glass/AFR700B) in an autoclave per the material manufacturers' recommended cure cycles. The number of panels and their stacking sequence depended upon the types of tests to be performed on the material as well as the number of specimens per test. Table 3 shows the various panels that were fabricated along with a description of each panel.

The quality of each panel was evaluated using non-destructive evaluations (NDE) and tag end testing. The various evaluations performed on the panels are shown in Table 4. The panels were then machined to produce the necessary test specimens to perform the test matrix shown in Table 2.

# 3.1 NON-DESTRUCTIVE EVALUATION (NDE)

Each panel was nondestructively evaluated before exposure or baseline testing using ultrasonic C-scans. The results from the panels are shown in Figures 1 and 2. The scale in the bottom of the figures indicates the percentage of returned signal. A 100% signal return signifies that no detectable damage/defects are present in the panel. Zero percent indicates no signal return and significant damage within the panel. Only the S-2/AFR700B panels exhibited any indication of damage/defects (as evidenced by the loss in returned signal). The damage/defects within the panels were shown to be a limited amount of microcracking as discussed in the Tag End Testing section of this report.

#### 3.2 TAG END TESTING

Each of the panels underwent a series of tag end tests. The tag end testing was used to screen panels for quality. Tag end testing included physical properties, cross-sections, grind down, and thermal analysis. The samples used for the tag end testing were removed from the panel in the location shown in Figures 3 thru 5. The methods and results of the tag end testing are described in sections 3.2.1 to 3.2.4.

# 3.2.1 PHYSICAL PROPERTIES

Four, 1" x 1" samples were removed from each panel. The thickness of each sample was measured with a micrometer. This thickness was then divided by the total number of plies to calculate the panel's per ply thickness. Each sample's density was measured in accordance with ASTM D792 "Standard Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement". The panel's density was taken as the average of the four samples from that particular panel. The samples taken from the AS4/3501-6 and IM7/5250-4 panels were then tested in accordance with ASTM D 3171 "Standard Test Method for Fiber Content on Resin-Matrix Composites by Matrix Digestion". The samples taken from the S-2/AFR700B panels were tested in accordance with ASTM D2584 "Standard Test Method for Ignition Loss of Cured Reinforced Resins". For both tests, the fiber volume, resin content and void volume were recorded for the panel from the average of the four samples from each panel. The results of the physical properties are shown in Table 5.

#### 3.2.2 CROSS-SECTIONS

One, 1" x 1" sample was removed from each panel. Care was taken to note the 0° direction of the sample. The sample was then mounted in a room temperature curing epoxy, polished and examined with an optical microscope. A representative photo was taken of each sample through its entire thickness. These photos are shown in Figures 6 thru 8. Examination of the cross-section included a verification of the ply stacking sequence and the panel's quality (void content, fiber distribution, microcracking, etc...). Results showed that Panel GB-IP-2 contained an incorrect ply stacking sequence over half of the panel in the diagonal direction this portion of the panel. This portion of the Panel GB-IP-2 was not used for mechanical testing, but was used for sandwich corrosion testing. Since the incorrect ply was located next to the center of the panel, the change in orientation of this one ply on the sandwich corrosion test results was judged as being minimal. The other half of the panel was correct. Each of the S-2/AFR700B panels exhibited a small amount of microcracking. These cross-sections were also used as baseline (not exposed) comparisons used in the testing described later in this report. Results are shown in Table 6.

#### **3.2.3 GRIND DOWN**

A 1" x 3" sample was removed from each panel being careful to note the 0° direction of the sample. One end of the sample was then sanded/polished at an angle of approximate 15° through the entire thickness of the sample. Examinations of the polished surface were used to verify the ply stacking sequence. All of the panels exhibited the correct layup except for GB-IP-2 (as discussed Section 3.2.2). Results are shown in Table 6.

#### 3.2.4 THERMAL ANALYSIS

One 1/8" x 1/8" sample was removed from each panel for thermal analysis testing. The sample was dried in an oven at 200° F until the sample weight loss was less than 0.01% over two consecutive days. The sample was tested with a thermal mechanical analyzer (TMA) in order to measure the panels glass transition temperature (Tg). The Tg was measured by identifying the onset point at which the coefficient of thermal expansion (CTE) significantly increased. Results are shown in Table 6.

#### 3.3 SPECIMEN PREPARATION

The necessary specimens were removed from the composite panels in the locations shown in Figures 3 thru 5. The specimens were machined further to meet the dimensional specifications of the various tests. The dimensional specifications for ASTM D3518/D3518M-94 "Standard Test Method for In-Plane Shear Response of Polymer Matrix Composite Materials by Tensile Test of a +/- 45° Laminate IPS (IPS) and Northrop Open-Hole Compression (OHC) test specimens are shown in Figure 9. The remaining test specimens were cut/machined to the nominal dimensions shown in Figures 3 thru 5. The test specimens were labeled according to material, test, and specimen number. The panel in which each of the specimens was removed is shown in Table 7. The specimen dimensions (width and thickness) were measured prior to deicer exposure.

#### 4.0 TESTING

Testing consisted of drying the specimens, exposing the specimens to deicers, and mechanical and physical testing. The test specimens were conditioned by drying them out prior to the deicer exposure. The "dry" weight of the specimens were measured. The specimens were then exposed to the deicer fluids. At the end of the exposure, the specimens were reweighed prior to testing. Two mechanical test methods were used; ASTM D3518/D3518M-94 and the Northrop Open-Hole Compression Test. Physical testing included Barcol hardness, thermal analysis, and microscopy. Corrosion effects were evaluated by using the sandwich corrosion test and measuring galvanic corrosion current flow. Thermal oxidative stability was evaluated for IM7/5250-4 and S-2/AFR700B by exposing the material to deicing fluid then holding at 400°F and 700°F respectively for 100 hours. After completion of the thermal exposure, the weight loss per surface area was measured.

# 4.1 SPECIMEN DRYING / DEICER FLUID EXPOSURE / WEIGHT GAIN

Test specimens were conditioned by drying them in a vacuum oven at 140-145°F and 25-30 inches of mercury vacuum over a weekend. After drying, the specimens were weighed then exposed by soaking under the deicer fluid for 4 hours then allowing to air dry at room temperature for 20 hours. This process was repeated for four additional days. The specimens were rinsed and allowed to remain at ambient conditions over the weekend. The exposed specimens were then weighed in order to calculate the total weigh gain. This gave the specimens a total of 20 hours submersed in deicing fluid over the course of a week. Out of each group of three mechanical test specimens one specimen was tested on Monday, the next specimen on Tuesday, and the last specimen on Wednesday. The physical test specimens were tested as soon as possible after deicer exposure.

#### 4.2 MECHANICAL TESTING

The mechanical testing performed on the specimens included IPS and OHC. The IPS tests were conducted at room temperature and elevated temperature conditions. The OHC specimens were tested at room temperature.

### 4.2.1 IN-PLANE SHEAR

Since the purpose of this effort was to evaluate the effect of deicing fluid exposure there were some concerns about the effect of deicing fluid exposure on strain gages or adhesively bonded tabs, especially for elevated temperature testing. The concern with strain gages or adhesively bonded tabs was that if bonding was done before exposure the deicers may weaken the adhesive bond. If bonding was done after exposure the absorbed deicer may prevent good surface adhesion. To eliminate the need to use tabs a Mechanical Testing Systems (MTS) machine with hydraulically actuated grips and surfalloy grip faces was used. The surfalloy grip faces provided an adequate gripping surface without severe grip serrations. A grip pressure of 1000 pounds per square inch (psi) was found to be adequate for AS4/3501-6 and IM7/5250-4. For the thicker S-2/AFR700B a grip pressure of 2000 psi was necessary. These levels of grip pressure were found to be high enough to prevent slippage and low enough so failures within the grips did not occur. The specimen grip area was roughly 1"x1". Crosshead movement was used for strain measurement. To get an accurate strain measurement with crosshead movement the specimen must not slip in the grips. ASTM D3518 defines the ultimate shear stress as the lesser of the maximum shear stress at failure or stress at 5% shear strain. The shear data given in this report is reported for 5% axial strain and for ultimate failure at any strain level, since an evaluation of changes due to the effect of absorbed deicers were desired. The relationship between axial strain and shear strain is given as:

 $\gamma_{12}=\varepsilon_x-\varepsilon_y$  where  $\gamma_{12}$  is the shear strain,  $\varepsilon_x$  is the axial strain and  $\varepsilon_y$  is the transverse strain.

For a uniaxial test, the shear strain is elevated over the longitudinal strain by the lateral contraction (transverse strain) of the specimen. The strain level reported in this report is consequently higher than that commonly used in D3518.

# 4.2.2 OPEN-HOLE COMPRESSION

The Northrop Open-Hole Compression Test Method was used for generating open-hole compression properties. This fixture uses an end-loaded side-supported specimen. The ultimate failure load was converted to a ultimate failure stress using the formula:

σ=P/wt where: σ=failure stress, P=failure load, w= width and t= thickness

Note this formula uses gross overall specimen dimensions and does not reduce the cross-sectional area due to the though-the-thickness hole.

# 4.3 PHYSICAL PROPERTY TESTING

The physical property testing performed included Barcol hardness, thermal analysis, cross-sectional analysis, sandwich corrosion, and thermal oxidative stability.

# **4.3.1 BARCOL HARDNESS**

Barcol hardness measurements were made with a Barcol hardness tester in accordance to ASTM D 2583 "Standard Test Method for Indentation Hardness of Rigid Plastics by Means of A Barcol Impressor". A total of five readings were performed on each sample's tool side (side of the panel fabricated on the tool). The average of the readings was recorded as the hardness of the sample.

#### 4.3.2 THERMAL ANALYSIS (Tg)

The specimens were dried in an oven at 200°F until less than 0.01% weight loss was recorded for two consecutive days. The material's Tg was measured with a thermal mechanical analyzer (TMA). The glass transition temperature (Tg) was measured as the onset temperature at which the material's coefficient of thermal expansion (CTE) changed significantly.

#### **4.3.3 CROSS SECTIONAL ANALYSIS**

The 1"x1" samples were mounted in epoxy, polished, and examined with an optical microscope. The surfaces of the samples were examined for evidence of damage caused by the deicing fluids.

#### **4.3.4 SANDWICH CORROSION**

The test was conducted in accordance to ASTM F 1110 "Standard Test Method for Sandwich Corrosion" with a couple of modifications. The test calls out placement of a 1" x 3" piece of saturated (with appropriate deicing fluid) filter paper between two pieces of 2024-T3 aluminum 2" x 4" in size. These sandwich constructions were placed into a 100° F oven for eight hours, removed and placed in a humidity chamber at 100°F/95-100% humidity for 16 hours. This cycle was repeated for a total of 5 days. At the end of the 5th day, the specimens were left in the humidity for an additional 48 hours. At the completion of the exposure, the sandwich structures were removed from the humidity, cleaned (removal of filter paper and fluid), and examined with a stereomicroscope for the severity of corrosion. Each aluminum specimen was then given a ranking based on the corrosion severity rating system given by the specification. The modifications to the specification included using the IM7/5250-4 composite material as one of the sandwich members. The second modification was that the aluminum surface was prepared by a thorough cleansing to remove any oils or surface contaminants.

#### **4.3.5 GALVANIC CORROSION**

For this study, the galvanic current between an Al 2024-T3 metal sample and an IM7/5250-4 composite sample was measured in various deicing fluids; UCAR, E-36, sodium acetate, urea, and sodium formate. For comparison, simulated seawater consisting of 0.6 molar sodium chloride solution was used. Figure 10 shows the galvanic corrosion cell. Each sample was taped to expose an area 2.54 cm by 2.54 cm (6.45 cm<sup>2</sup>) on the front and back face of the samples and 2.54 cm by 0.32 cm (0.82 cm<sup>2</sup>) for each of the three exposed edges. A ¼ inch rubber spacer was used to separate the samples. A rubber band over the tape was used to hold the galvanic couple together. Silver filled conductive paint was used on the composite sample to establish electrical contact between the cut Graphite fiber ends and the lead to the

potentiostat. The sample pairs were placed into a 100 ml beaker. The beaker was then filled so that the liquid level was higher than the lower edge of the upper protective tape layer.

The galvanic currents were measured using an EG&G PARR 273A potentiostat connected to multiple samples through a 314 multiplexer. Two electrode measurements were made by connecting the reference and counter electrodes to the aluminum sample and the working electrode to the composite. A negative current measured by the working electrode indicates reduction of that electrode while a positive current would indicate oxidation of the electrode.

Current was applied to the composite and aluminum plates to keep the potential difference at zero volts. This is equivalent to shorting the samples together. This applied current was the same as the galvanic current measured through a theoretical zero resistance wire short circuit. Each galvanic cell was measured for 20 minutes. After all six of the sample pairs were monitored, the measurement cycle was reinitiated. For the second and each successive cycle, the galvanic current was measured for only 5 minutes. Between the second and third cycle and each successive cycle, there was a two hour pause in data collection. The voltage between the samples in each sample pair remained at 0 volts throughout the experiment.

# 4.3.6 THERMAL OXIDATIVE STABILITY

Thermal oxidative stability (TOS) testing was performed on ten 1" x 1" samples for each of the deicing exposures on IM7/5250-4 and S-2/AFR700B materials. The samples were cleaned with Scotchbrite and soapy water. Each sample was cleaned in an ultrasonic bath (water medium). Upon removal of the samples, they were handled throughout the remainder of the testing with rubber gloves. The sample's length and width were measured. These measurements were used to calculate the total surface area of the sample. The surface area included both surfaces of the sample. The samples were weighed to x.xxxx grams. The samples were placed in a vacuum oven set at 200°F. A small fraction of these specimens were then weighed periodically until less than 0.01% weight loss was recorded for consecutive days. The weighed specimens were used to represent the entire batch of TOS specimens. After drying each sample was weighed. These weights were recorded as the "dry weights". The samples were placed on an oven rack that was covered with a fiberglass breather cloth. The samples were covered lightly with another layer of fiberglass breather cloth. A thermocouple was placed near the samples to monitor the temperature of the oven. The oven itself was a convection oven with the damper mostly closed (not air tight). The oven was heated to the desired temperature (400°F for IM7/5250-4 and 700°F for S-2/AFR700B) at 10°F/min. The samples were held at temperature for 100 hours. After 100 hours, the oven was returned to ~150°F. The samples were removed, placed into a desiccator (until they reached room temperature), and weighed. This final weight was subtracted from the "dry weight" to calculate the weight loss due to the thermal exposure. The weight loss was divided by the total surface area to achieve the weight loss per surface area (mg/cm<sup>2</sup>).

#### 5.0 RESULTS

The results from the weight gain, mechanical testing and physical testing were summarized. Due to the small number of specimens performed per test/material/deicer fluid, the data generated in this program is for screening purposes only and cannot be used to generate statistically significant engineering design data.

# **5.1 WEIGHT GAIN**

Table 8 is a summary of the test specimen weight gain when exposed to runway deicers. The weight gain was largest for S-2/AFR700B followed by IM7/5250-4 and AS4/3501-6. Exposure to 15% Octagon RD1431 SA resulted in the largest weight gain for both AS4/3501-6 and IM7/5250-4. Exposure to Cryotech E-36 resulted in the largest weight gain for S-2/AFR700B.

#### **5.2 MECHANICAL TESTING**

#### **5.2.1 IN-PLANE SHEAR**

AS4/3501-6 in-plane shear test data at 5% axial strain tested at ambient conditions is shown in Figure 11. Plotted is the average of the data. The error bars are created by adding and subtracting one standard deviation. Note the plot uses an expanded "y" scale to emphasize any differences. The error bars overlapped for the dried, 15% urea and 15% Octagon RD1431 SA exposed specimens. Other specimens had higher in-plane shear strength. When comparing E-36 to the liquid control UCAR PM-5197 the error bars also overlapped. Comparing the 15% solution of solid deicers, 15% Octagon RD1431 SA produced lower strength than the solid control (15% urea). Figure 12 illustrates the results of the in-plane shear test ran to ultimate failure. Note all the deicer solution exposed specimens had higher ultimate strengths than the dried specimens. Also note that material exposed to 15% Octagon RD1431 SA produced a higher ultimate strength than the solid control (15% urea). A summary of the results for the AS4/3501-6 material is shown in Table 9. The individual specimen results are shown in Appendix A.

IM7/5250-4 in-plane shear results at 5% axial strain tested under ambient conditions are shown in Figure 13. The only difference that can be noted about this graph is that the Cryotech E-36 is slightly above the dried specimen error bars. Note that the E-36 data has an exceptionally small error bar, possibly a result of only running three specimens thus not adequately assessing the standard deviation of a larger population. Figure 14 shows the IM7/5250-4 in-plane shear results tested at ambient to ultimate failure. The dried control had a lower ultimate strength than specimens exposed to E-36 and all of the solid runway deicers. When comparing in-plane shear strength for the liquid and solid deicers only the 15% Octagon RD1431 SA is higher than the solid control (15% Urea). Figure 15 shows the results of in-plane shear testing at 5% axial strain and 350°F for IM7/5250-4. The dried specimens were higher than all the deicer exposed specimens except for UCAR PM-5197. Also the specimens exposed to 15% Octagon RD1431 SA have lower strengths than the solid control (15% urea). Figure 16 shows the ultimate shear strength at 350°F. Error bars for the controls and data sets overlapped. A summary of the test results for the IM7/5250-4 material are shown in Table 9. The individual specimen results are given in Appendix A.

The results for S-2/AFR700B at ambient conditions and 5% axial strain are shown in Figure 17. The dried specimens exhibited a higher strength than all except the UCAR PM-5197 exposed specimens. The liquid control UCAR PM-5197 is also higher than the Cryotech E-36. The solid control has a greater strength than the other two solid deicers 15% Safeway SF and 15% Octagon RD1431 SA. The ultimate strength results at ambient are shown in Figure 18. The results from the 15% urea exposed specimens have slightly higher strengths than the dried material. Specimens exposed to Cryotech E-36 and 15% Octagon RD1431 SA have lower strengths than the dried material. The liquid control UCAR PM-5197 specimens have higher strength than the Cryotech E-36 exposed specimens. The solid control 15% urea exposed specimens have higher strength than both of the other solid deicer exposed specimen groups. Figure 19 shows the results for S-2/AFR700B at 550°F and 5% axial elongation. The dried strength is higher than all the other groups except UCAR PM-5197. The UCAR PM-5197 is also greater strength than the other liquid deicer Cryotech E-36. The control solid deicer 15% urea had greater strength than both the 15% Safeway SF and 15% Octagon RD1431 SA. Figure 20 shows the results of in-plane shear testing for S-2/AFR700B to ultimate failure at 550°F. The dried specimens have greater strength than the Cryotech E-36 exposed specimens. The Cryotech E-36 exposed specimens also had a lower strength than the liquid control UCAR PM-5197. No difference beyond one standard deviation was seen in the strength of solid deicer exposed specimens. A summary of the test results for the S-2/AFR700 material are shown in Table 9. The individual specimen test results are given in Appendix A.

#### **5.2.2 OPEN-HOLE COMPRESSION**

The results for AS4/3501-6 graphite/epoxy tested at ambient conditions are shown in Figure 21. Like the in-plane shear results the average with the error bars being one standard deviation above and below the mean are plotted. No differences greater than the error bars was noted for AS4/3501-6. This data also includes two additional groups of specimens "ambient" and "distilled water". The "ambient" group was tested without drying or exposure to fluids. The "distilled water" group was dried and exposed to distilled water using a cycle the same as for the deicers. A summary of the open-hole test results for AS4/3501-6 are listed in Table 10. The individual specimen results are given in Appendix C.

A plot of the results for IM7/5250-4 graphite/BMI is shown in Figure 22. The dried specimen controls exhibited lower strength than all of the deicer fluid exposed specimen groups except 15% urea and 15% Safeway SF exposed groups. The liquid control was slightly higher than the Cryotech E-36 exposed material. No difference existed beyond the error bars on the solid deicer exposed materials. A summary of the IM7/5250-4 open-hole compression results is given in Table 10. The individual specimen results are given in Appendix C.

Figure 23 shows the open-hole compression strength results for S-2/AFR700B. Open-hole compression strengths for the dried specimens were higher than all deicer exposed groups. The liquid control group UCAR PM-5197 had higher strength than the Cryotech E-36 exposed group. The solid deicer control group, 15% urea, had higher strength than 15% Safeway SF and 15% Octagon RD1431 SA. A summary of the S-2/AFR700B open-hole compression results is given in Table 10. The individual specimen results are given in Appendix C.

#### **5.3 PHYSICAL TESTING**

#### 5.3.1 BARCOL HARDNESS

The results are shown in Table 11. The results showed very little effect from the exposures to any of the specimens. The only specimen that did show a small amount of difference was the S-2/AFR700B material exposed to the Cryotech E-36 (potassium acetate).

#### 5.3.2 THERMAL ANALYSIS (Tg)

The results are shown in Table 11. Results showed that only the S-2/AFR700B material exposed to the 15% Urea demonstrated any significant effect on the Tg, as compared to the non-exposed (dried) samples.

#### 5.3.3 CROSS SECTIONAL ANALYSIS

Results of the examinations are shown in Table 11. Results showed that no significant damage was incurred by any of the materials.

#### **5.3.4 SANDWICH CORROSION**

Results are shown in Table 12. None of the deicer fluids appeared to have any significant effect on the aluminum. Examination of the composite's surface with a scanning electron microscope revealed surface cracking in the outer layer of resin, see Figure 24. This cracking was only observed on the side of the composite which was mated with the aluminum during exposure. The observed surface cracking was independent of whether the tool or bag side of the composite was mated with the aluminum.

### **5.3.5 GALVANIC CORROSION**

All currents measured were negative indicating the aluminum was the anodic component of the couple and the composite was the cathodic component. The raw data was divided by the exposed surface area of the aluminum sample. The absolute value of this number is displayed and represents the anodic current density (A/cm²) resulting from degradation of the aluminum sample.

Higher values of current density represent a higher galvanic corrosion rate. Severity of the galvanic current density as a function of deicing fluid is as follows: sodium chloride > sodium acetate > sodium formate > UCAR > urea > E-36. Figure 25 graphically shows galvanic current drop versus time. Only the aluminum sample in the sodium chloride showed signs of corrosion. In the sodium chloride solution, pitting of the aluminum was observed. However, the galvanic currents were extremely small.

It should be remembered that a galvanic corrosion rate is not only a function of surface interface properties of the material but also of the solution resistance. In other words, if the samples in this study were closer than ¼ inch, the galvanic corrosion rates would have been higher. In addition, if the specimens were in intimate contact, the possibility of forming a dissimilar metal crevice exists. Under these conditions, galvanic behavior becomes much more complex and the simple galvanic current tests performed here may incorrectly predict the galvanic corrosion behavior of the materials studied.

#### 5.3.6 THERMAL OXIDATIVE STABILITY

Figures 26 and 27 show the results of thermal oxidative stability (TOS) testing for IM7/5250-4 and S-2/AFR700B materials, respectively. Similar to the plots for mechanical testing, the TOS results plotted are the average with the error bar being one standard deviation above and below the mean.

Each of the deicer fluids caused an increase in weight loss/surface area than the dried (no exposure) specimens. In particular, the UCAR PM5197 caused the greatest difference in the IM7/5250-4 composite material while the Cryotech E-36 caused the greatest difference in the S-2/AFR700B composite material.

#### **6.0 CONCLUSIONS:**

This report has examined the effect of environmental friendly runway deicers on the mechanical and physical properties of some commonly used composite materials. Several differences have been noted between dried specimens and the deicer exposed material. Also the liquid and solid deicer results were compared with the liquid and solid control deicers respectively.

The weight gain due to deicer exposure was largest for S-2/AFR700B followed by IM7/5250-4 and AS4/3501-6. Exposure to 15% Octagon RD1431 SA resulted in the largest weight gain for AS4/3501-6 and IM7/5250-4. Exposure to Cryotech E-36 resulted in the largest weight gain for S-2/AFR700B.

In-plane shear testing for AS4/3501-6 showed that the dried group exhibited lower strength than deicer exposed groups. All deicer exposed groups exhibited similar behavior except for the Octagon RD 1431 SA exposed group which exhibited a more ductile behavior and higher ultimate strength. In-plane shear testing of IM7/5250-4 at ambient temperature demonstrated that the UCAR PM-5197 exposed group exhibited failure at low strain and strength levels and the Octagon RD 1431 SA exposed group exhibited a ductile behavior with higher ultimate strength. IM7/5250-4 tested at 350°F showed that the 15% Octagon RD 1431 SA exposed group exhibited greater ductility and lower strength at the 5% axial strain level. In-plane shear testing of S-2/AFR700B specimens at ambient demonstrated that the Cryotech E-36 and 15% Octagon RD 1431 SA exposed groups had lower strength at 5% axial strain and lower ultimate strength than the other deicer exposed groups. In-plane shear testing of S-2/AFR700B at 550°F demonstrated that the Octagon RD 1431 SA exposed group had lower strength at 5% axial strength and the Cryotech E-36 exposed group had a lower ultimate strength.

Open-hole compression testing of AS4/3501-6 demonstrated no significant effect of deicer exposure. For IM7/5250-4 the dried group produced the lower strength than the deicer exposed groups. Open-hole compression results for S-2/AFR700B demonstrated that all deicer exposed groups had significantly lower strength than the dried group, the largest decrease was 38% for the Cryotech E-36 exposed group.

The Barcol hardness changed very little after exposure to deicers. The only specimen that did show a small amount of difference was the S-2/AFR700B material exposed to the Cryotech E-36. Measured glass transition temperatures (Tg) showed that only the S-2/AFR700B material exposed to the 15% Urea demonstrated any effect, as compared to the non-exposed (dried) samples. Cross-sectional analysis noted no significant damage for any group.

Sandwich corrosion noted very slight corrosion of the aluminum side and surface cracking of the composite material side. Galvanic current was measured for IM7/5250-4 and 2024-T3, the galvanic current density as a function of deicing fluid is as follows: sodium chloride > sodium acetate > sodium formate > UCAR > urea > E-36.

Finally thermal oxidative stability testing showed that each of the deicer fluids caused an increase in weight loss/surface area than the dried (no exposure) specimens. In particular, the UCAR PM5197 caused the greatest difference in the IM7/5250-4 composite material while the Cryotech E-36 caused the greatest difference in the S-2/AFR700B composite material.

#### 7.0 RECOMMENDATIONS:

When new deicers formulations are developed they should be evaluated against the database established in this report.

#### 8.0 SPECIAL RECOGNITION:

Special recognition is given Brad Pinnell of the University of Dayton Research Institute (UDRI) who arranged the production of panels and specimen preparation. Brad also performed tag end testing, cross-sections, grind down analysis, thermal analysis, and thermal oxidative stability testing. John Eblin of UDRI performed in-plane shear and open-hole compression testing. Jim Dante of the University of Dayton Research Institute conducted the galvanic corrosion testing. Dan Laufersweiler of United Technologies Corporation conducted ultrasonic C-scans of the panel. Finally, Mike Stark of the Southwestern Ohio Council on Higher Education (SOCHE) prepared the comparison stress-strain curves shown in Appendix B and D.

# PREPARED BY:

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<u>PUBLICATION REVIEW</u>: This report has been reviewed and approved.

SHUI-NAN CHUANG, Chief Acquisition Systems Support Branch Systems Support Division Materials and Manufacturing Directorate

cc:

AFRL/MLSA AFRL/MLSC AFRL/MLSC (L. Gulley)

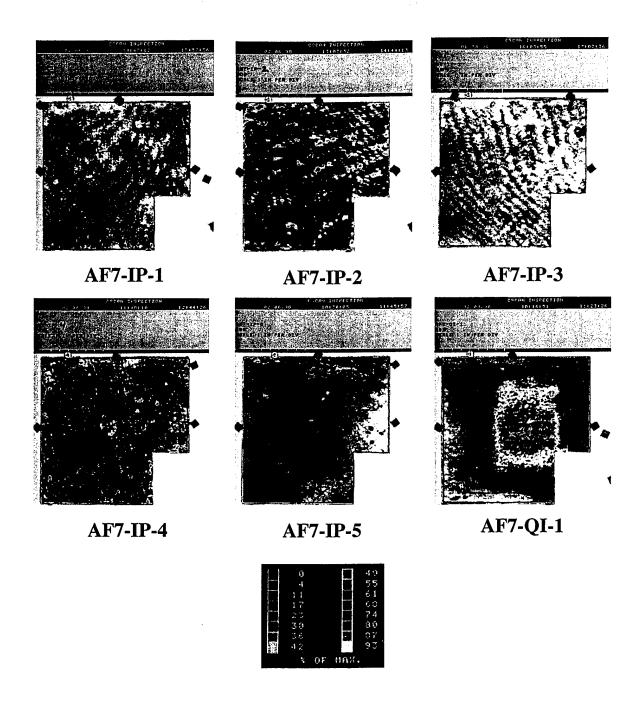


Figure 1. NDE C-scans of the S-2/AFR700B panels. The tag end specimens were removed (bottom right corner) from each panel prior to NDE testing. The loss in signal was attributed to microcracking within the panels.

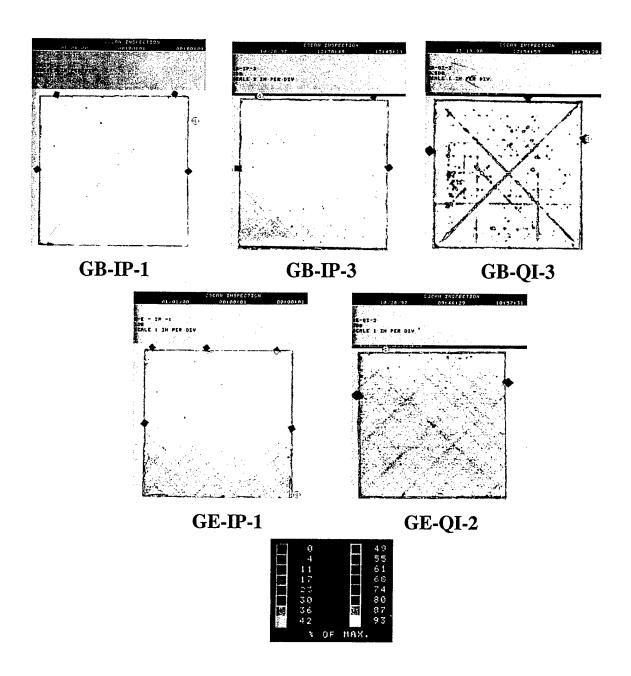


Figure 2. NDE C-scans of the IM7/5250-4 and AS4/3501-6 panels. The C-scan for panel GB-IP-2 was unavailable.

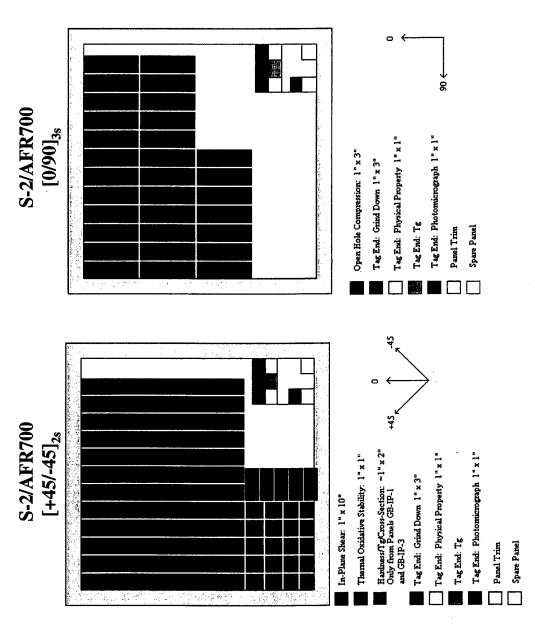


Figure 3. Schematic drawing of the S-2/AFR700B panels and the locations in which the test and tag end specimens were removed.

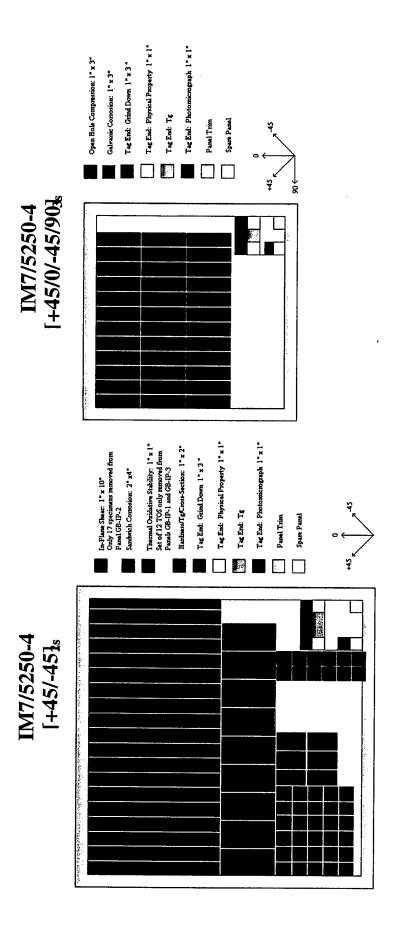


Figure 4. Schematic drawing of the IM7/5250-4 panels and the location in which the test and tag end specimens were removed.

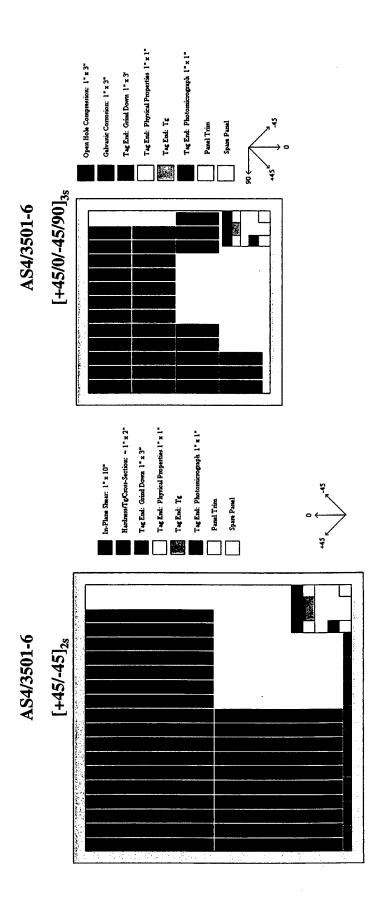


Figure 5. Schematic drawing of the AS4/3501-6 panels and the location in which the test and tag end specimens were removed.

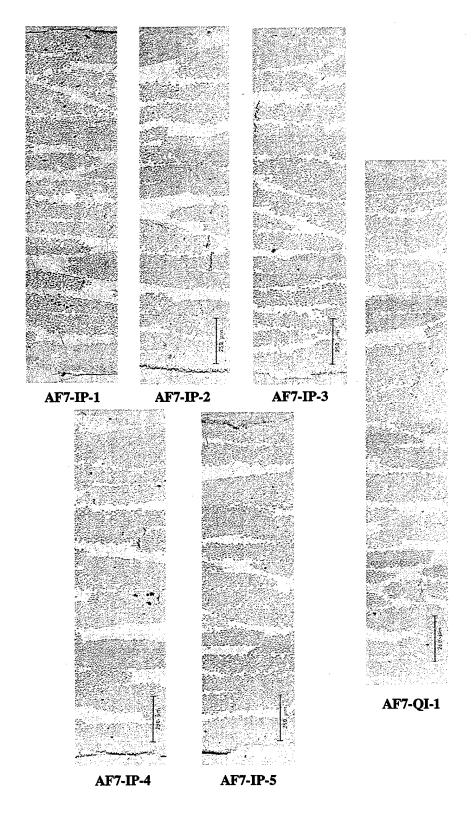


Figure 6. Cross-sectional photomicrographs of the S-2/AFR700 Tag End cross-sections. Small amounts of microcracking were observed. Scale bar in photos equals 250 microns.

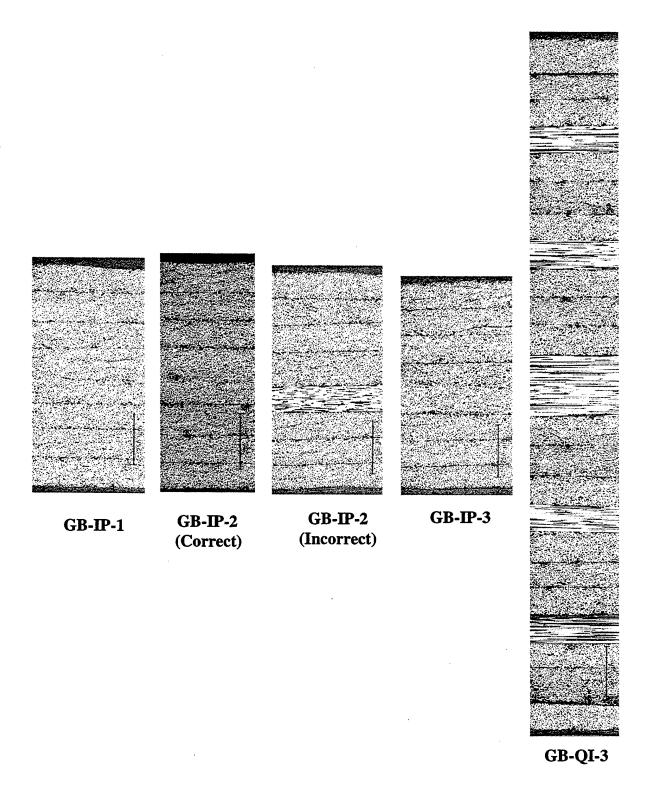


Figure 7. Cross-sectional photomicrographs of the IM7/5250-4 Tag End cross-sections. One half of panel GB-IP-2 contained an incorrect layup while the other half was correct. Scale bar in photos equals 250 microns.

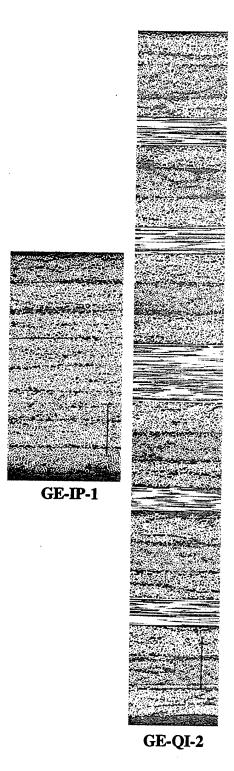


Figure 8. Cross-sectional photomicrographs of the AS4/3501-6 Tag End cross-sections. Scale bar in photos equals 250 microns.

Figure 9. Drawings of the Open-Hole Compression and In-Plane Shear test specimens.

Notes for Open Hole Compression Specimen:

1.000" +/- 0.005"

Notes for Open Hole Compression Specimen:

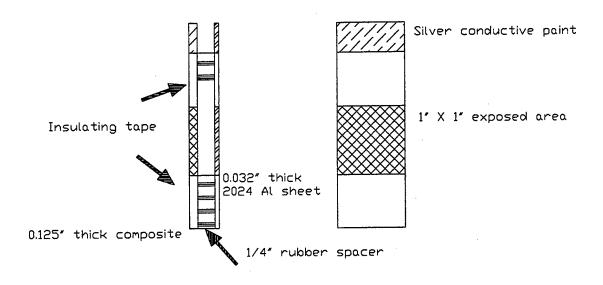
(1) Top and bottom surfaces shall be flat and parallel to 0.0005"

(2) Specimen thickness shall vary by no more than 0.010" along the length from nominal

(3) Specimen longitudinal edges shall be parallel to +/- 0.002"

(4) All machined specimen dimensions shall be within +/- 0.010" from nominal

(5) Hole shall be centered by length and width and shall be within +/- 0.005" from nominal



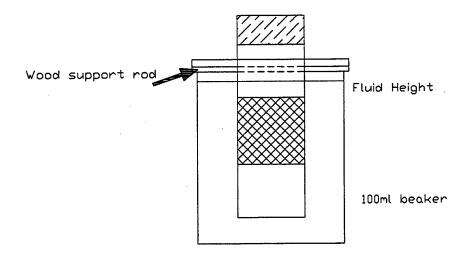


Figure 10. Galvanic Corrosion Cell

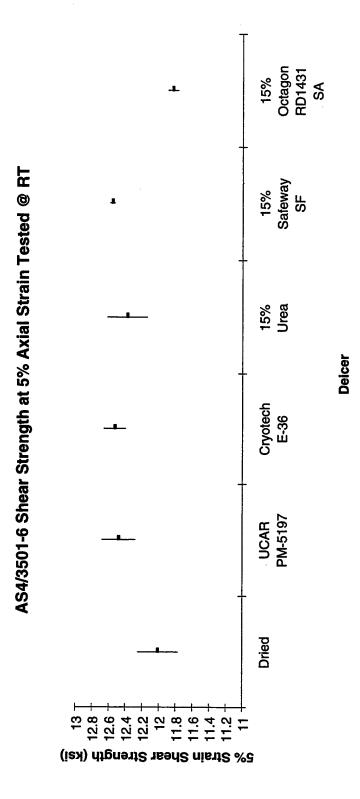


Figure 11. Plot of the AS4/3501-6, IPS testing (5% strain) at RT.

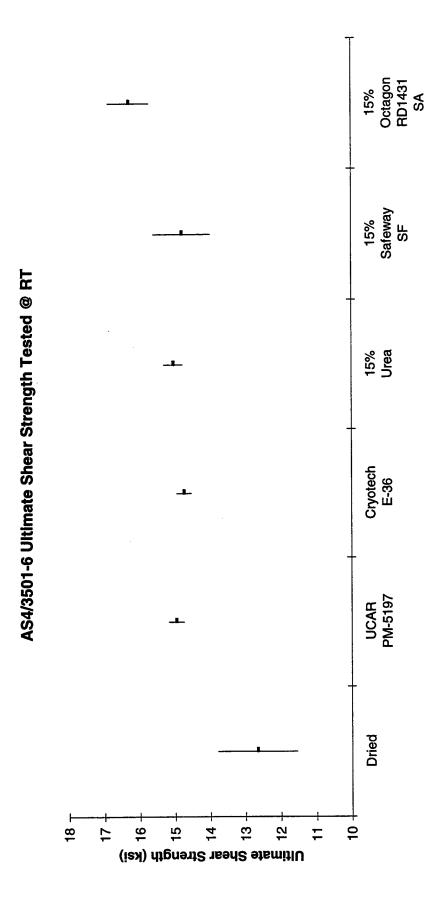


Figure 12. Plot of the AS4/3501-6, IPS testing (ultimate) at RT.

Deicer

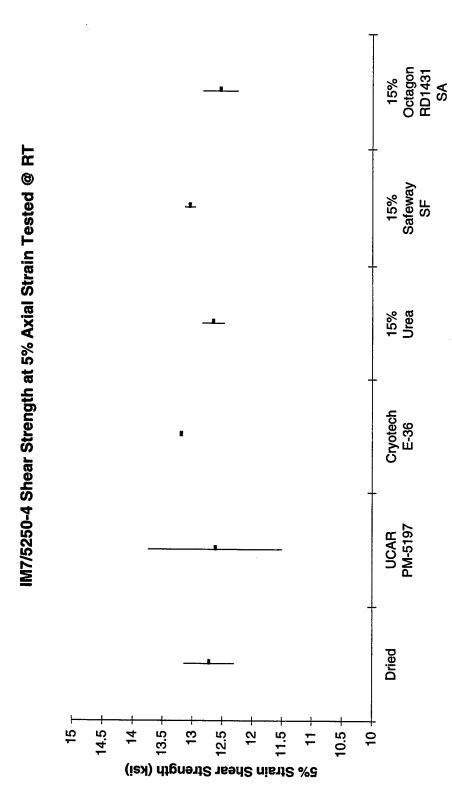


Figure 13. Plot of the IM7/5250-4, IPS testing (5% strain) at RT.

Deicer

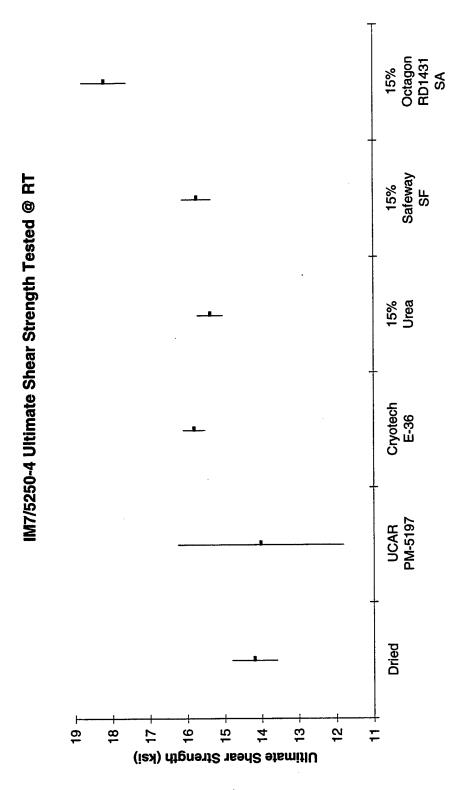


Figure 14. Plot of the IM7/5250-4, IPS testing (ultimate) at RT.

**Deicing Fluid** 



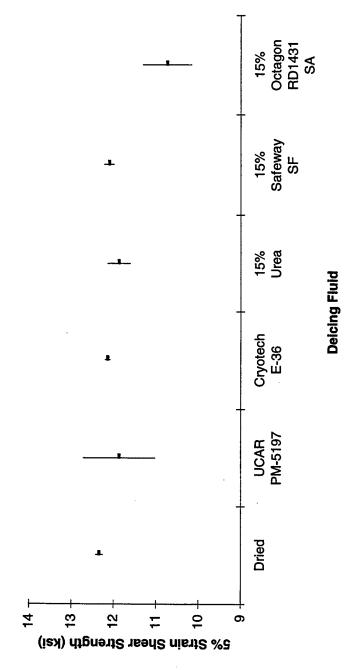


Figure 15. Plot of the IM7/5250-4, IPS testing (5% strain) at 350°F.



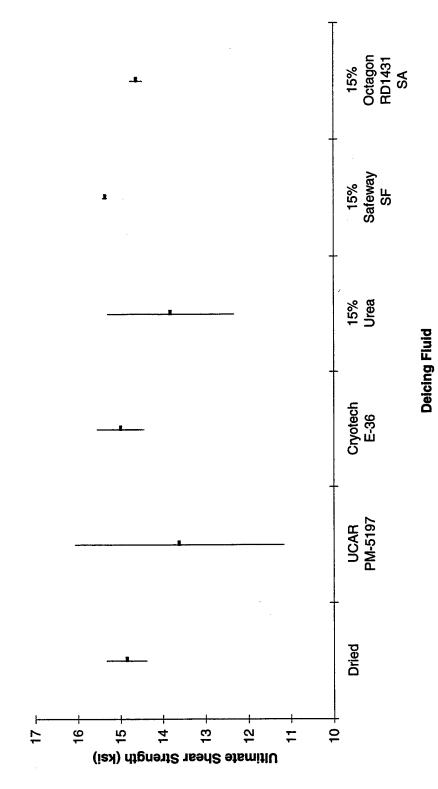


Figure 16. Plot of the IM7/5250-4, IPS testing (ultimate) at 350°F.

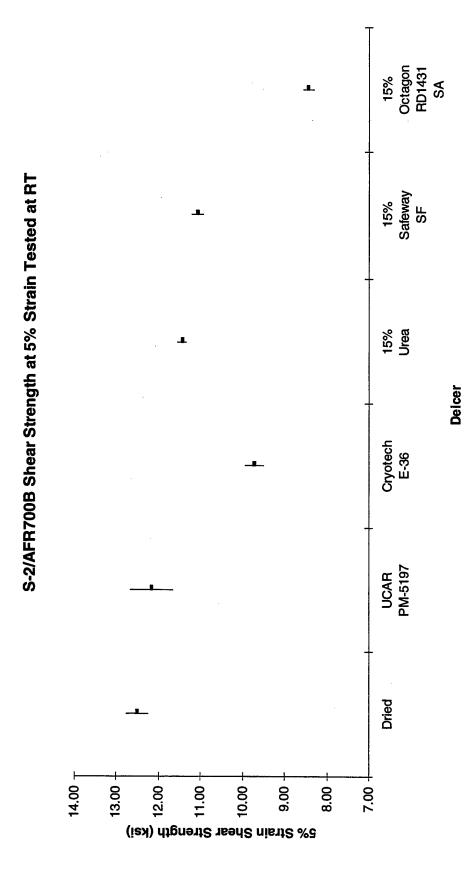


Figure 17. Plot of the S-2/AFR700B, IPS testing (5% strain) at RT.



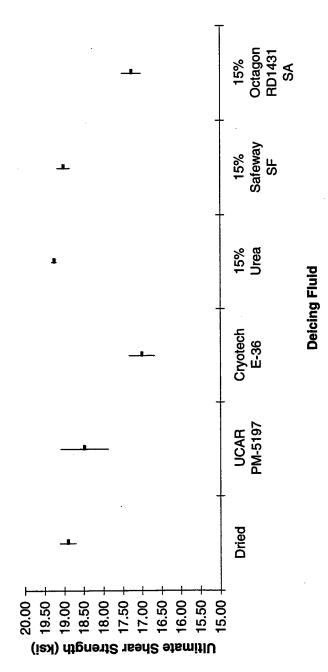


Figure 18. Plot of the S-2/AFR700B, IPS testing (ultimate) at RT.

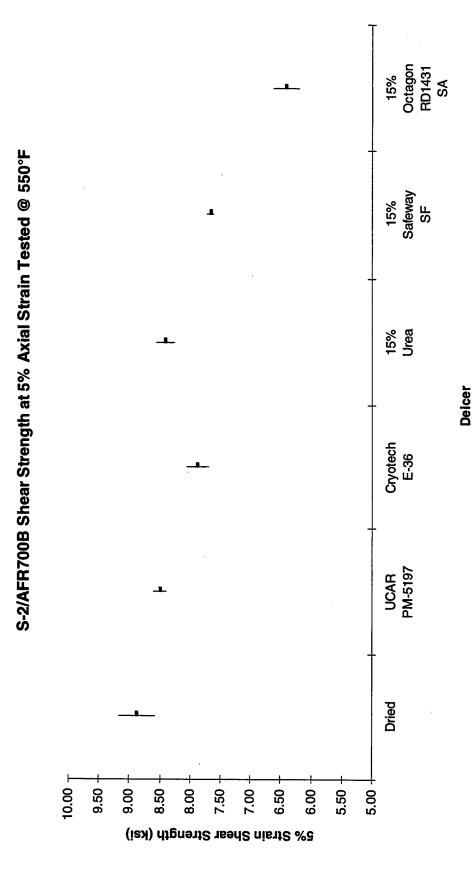


Figure 19. Plot of the S-2/AFR700B, IPS testing (5% strain) at 550°F.



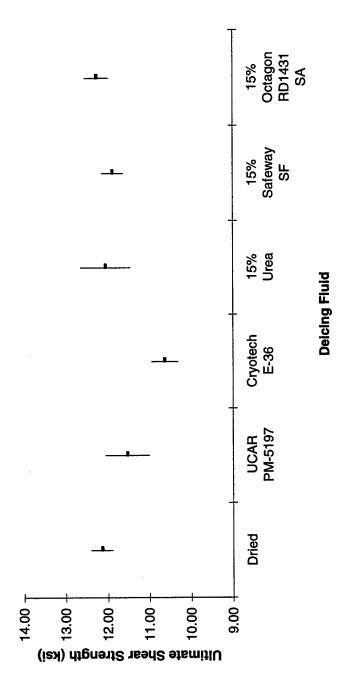


Figure 20. Plot of the S-2/AFR700B, IPS testing (ultimate) at 550°F.

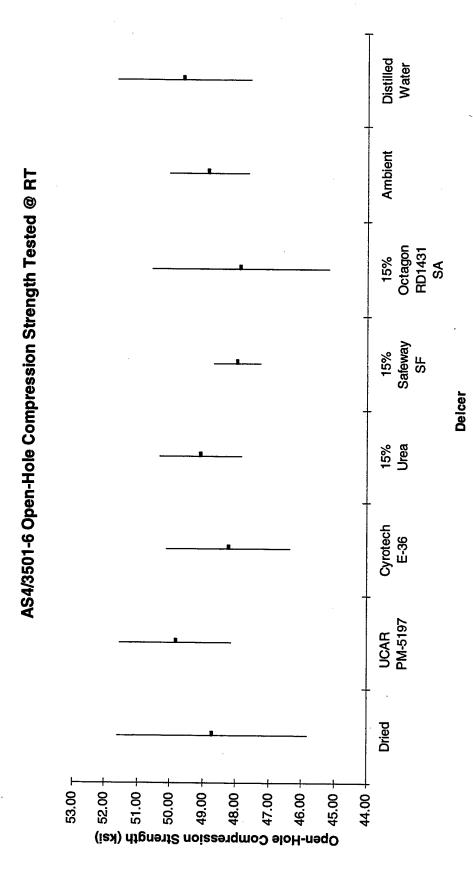


Figure 21. Plot of the AS4/3501-6, OHC testing at RT.



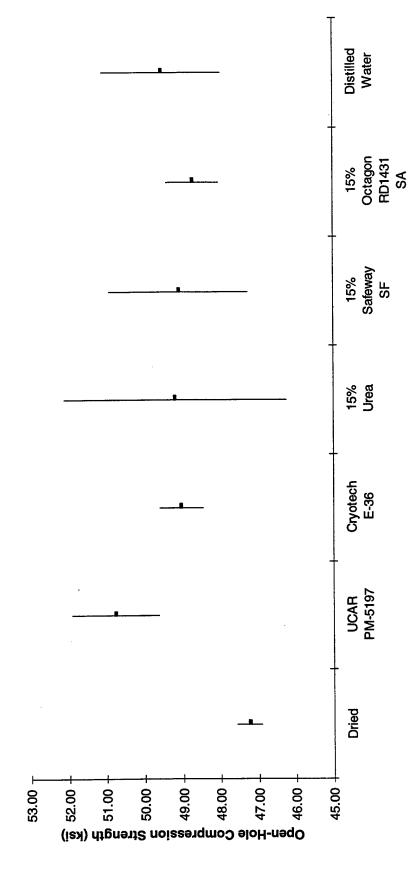


Figure 22. Plot of the IM7/5250-4, OHC testing at RT.

Deicer

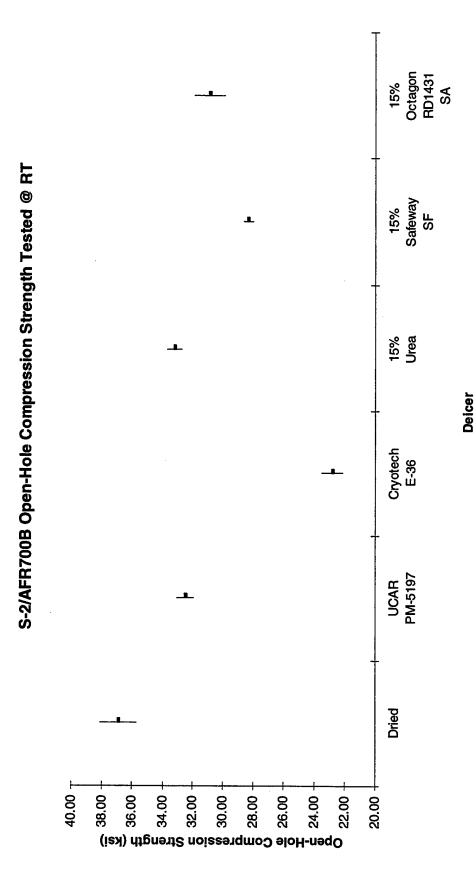


Figure 23. Plot of the S-2/AFR700B, OHC testing at RT.

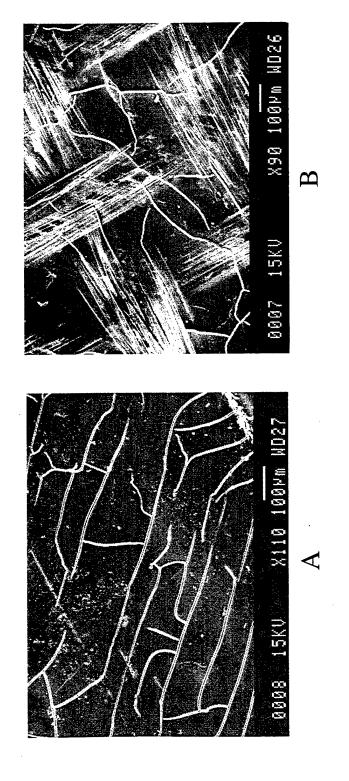


Figure 24. SEM photos of the IM7/5250-4 surfaces (A: tool side; B: bag side) exposed to the UREA deice fluid (sandwich corrosion). Cracking in the outer layer (surface) of the resin was observed.



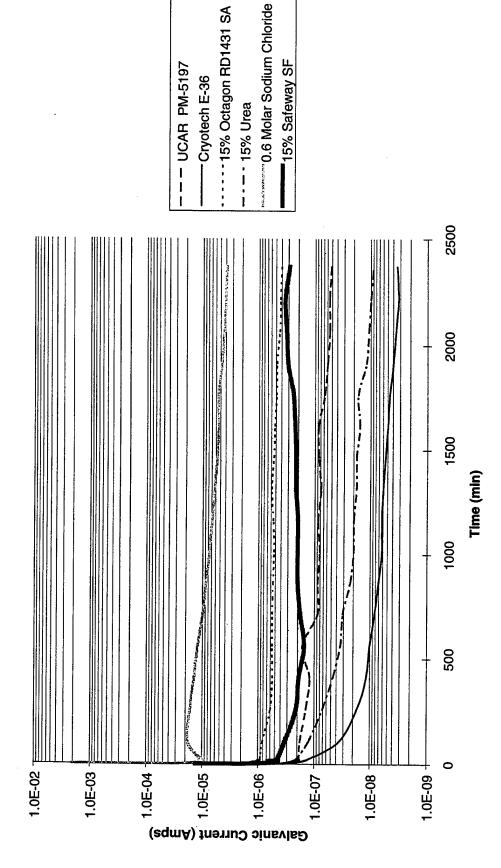
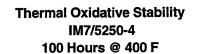


Figure 25. Galvanic Current for IM7/5250-4 and 2024-T3 in Deicers



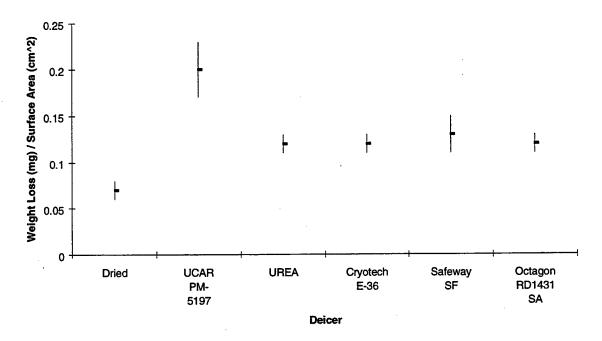


Figure 26. Results from the thermal oxidative stability testing of the IM7/5250-4.

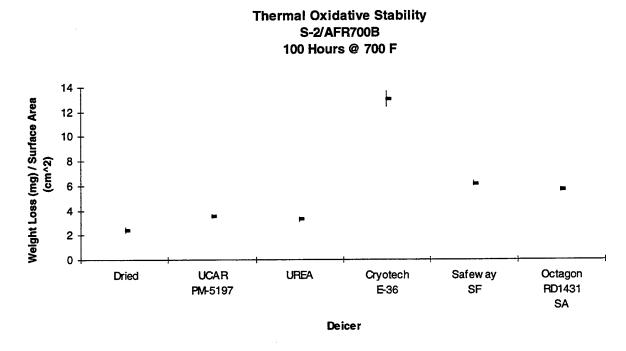


Figure 27. Results from the thermal oxidative stability testing of the S-2/AFR700B.

**TABLE 1** 

Summary of Deicer Fluids Studied in the Investigation

	Deicer Compound		
Designation	Manufacturer	Solid / Liquid	Constituents
None	Not Applicable (N/A)	N/A	N/A
UCAR PM-5197	Union Carbide Corporation	Liquid	50% Ethylene Glycol, 23% Urea 25% Water, and Corrosion Inhibitors
Cryotech E-36	Cryotech	Liquid	50% Potassium Acetate 50% Water, and <1% Corrosion Inhibitors
UREA	Agricultural Minerals Corporation	Solid	97-99% Urea
Safeway SF	Clariant (Canada) Incorporated	Solid	Minimum 98% Sodium Formate Corrosion Inhibitors
Octagon RD1431 SA	Octagon Process Incorporated	Solid	<80% Sodium Acetate Trihydrate

**TABLE 2** 

Summary of Tests Performed and the Number of Specimens Per Test on the Composite Materials

		In-Plan	In-Plane Shear			Galvanic	Galvanic Sandwich	Barcol		Cross-
Material	Layup Orientation	RT	ET <sup>(1)</sup>	OHC <sup>[2]</sup>	TOS <sup>[3]</sup>	Corrosion (	Corrosion	Corrosion Hardness	Tg	Section
AS4 / 3501-6	[+45/0/-45/90] <sub>3s</sub>	N/A <sup>[4]</sup>	N/A	3	N/A	N/A	N/A	N/A	N/A	N/A
AS4 / 3501-6	[+45/-45] <sub>2s</sub>	က	N/A	N/A	Z/A	N/A	4	-	-	-
									**************************************	
IM7 / 5250-4	$[+45/0/-45/90]_{3s}$	N/A	N/A	က	N/A	-	N/A	N/A	NA	N/A
IM7 / 5250-4	[+45/-45] <sub>2s</sub>	က	က	N/A	10	N/A	4	-	-	<b>-</b>
S-2 / AFR700B	se[06/0]	N/A	ĕ N N	ო	ΑN	ΑŅ	N/A	N/A	N/A	N/A
S-2 / AFR700B	[+45/-45] <sub>2s</sub>	က	က	A/N	10	N/A	4	-	<b></b>	-
<sup>[1]</sup> IM7 / 5250-4:	350°F		14 Not Applicable	licable						

550°F S-2 / AFR700B:

<sup>[2]</sup> Open-Hole Compression @ Ambient Only <sup>[3]</sup> Thermal Oxidative Stability

## **TABLE 3**Composite Panel Descriptions

Material	Material Description	Panel ID	# of Plies	Layup Orientation
AS4 / 3501-6	Graphite (Unidirectional) / Epoxy	GE-IP-1	8	[+45/-45] <sub>2s</sub>
AS4 / 3501-6	Graphite (Unidirectional) / Epoxy	GE-QI-2	24	$[+45/0/-45/90]_{3s}$
IM7 / 5250-4	Graphite (Unidirectional) / BMI	GB-IP-1	8	[+45/-45] <sub>2s</sub>
IM7 / 5250-4	Graphite (Unidirectional) / BMI	GB-IP-2	<b>©</b>	$[+45/-45]_{2s}$
IM7 / 5250-4	Graphite (Unidirectional) / BMI	GB-IP-3	80	[+45/-45] <sub>2s</sub>
IM7 / 5250-4	Graphite (Unidirectional) / BMI	GB-QI-3	24	$[+45/0/-45/90]_{3s}$
And the second s				
S-2 / AFR700B	Glass (Woven) / Polyimide	AF7-IP-1	8	[+45/-45] <sub>2s</sub>
S-2 / AFR700B	Glass (Woven) / Polyimide	AF7-IP-2	80	$[+45/-45]_{2s}$
S-2 / AFR700B	Glass (Woven) / Polyimide	AF7-1P-3	80	[+45/-45] <sub>2s</sub>
S-2 / AFR700B	Glass (Woven) / Polyimide	AF7-IP-4	80	$[+45/-45]_{2s}$
S-2 / AFR700B	Glass (Woven) / Polyimide	AF7-IP-5	80	$[+45/-45]_{2s}$
S-2 / AFR700B	Glass (Woven) / Polyimide	AF7-QI-1	12	$[0/90]_{3s}$

## **TABLE 4**Quality Tests per Panel

Test	# of Specimens	Measurements
Physical Properties	4	Fiber % by Volume, Matrix % by Weight,
		Void % by Volume, Panel Thickness,
		Per Ply Thickness, Density
Cross-Section		Panel Quality
Grind Down	-	Layup Orientation
Thermal Analysis	-	Тд
Non-Destructive Evaluation	1	Panel Quality

**TABLE 5** 

Composite Panel Physical Properties

Thickness Thickness GB-IP-1 0.0407 GB-IP-2 0.1303 GB-IP-2 0.0388 GB-IP-3 0.0400	(in) 0.0407 0.0388 0.0388		(g/cc) 1.595 1.572 1.572 1.576	+ <del>2</del>	Volume <sup>1</sup> Content <sup>2</sup> (% by vol) (% by vol) 63.11 0.19 59.22 0.47	Content <sup>2</sup> (% by vol) 0.19 0.47
GE-IP-1 0.0407 (in) GE-IP-1 0.0407 (GB-IP-1 0.0401 (GB-IP-2 0.0388 (GB-IP-3 0.0400 (GB-IP-3 0.	0.0401 0.0388 0.0388	(in) 0.0051 0.0054 0.0050 0.0049	(g/cc) 1.595 1.572 1.576 1.592	(% by wt) 29.21 32.57	(% by vol) 63.11 59.22	(% by vol) 0.19 0.47
GE-IP-1 0.0407 (GE-QI-2 0.1303 (GB-IP-1 0.0401 (GB-IP-2 0.0388 (GB-IP-3 0.0400	0.0407 0.1303 0.0388 0.0388	0.0051 0.0054 0.0050 0.0049	1.595 1.572 1.576 1.592	32.57	63.11 59.22	0.19
GE-QI-2 0.1303 (GB-IP-1 0.0401 (GB-IP-2 0.0388 (GB-IP-3 0.0400	0.0401 0.0388	0.0054	1.572 1.576 1.592	32.57	59.22	0.47
GB-IP-1 0.0401 (GB-IP-2 0.0388 (GB-IP-3 0.0400	0.0401	0.0050	1.576 1.592	79.00		
GB-IP-1 0.0401 (GB-IP-3 0.0400	0.0401	0.0050	1.576 1.592	79 00		THE STATE OF THE S
GB-IP-2 0.0388 (GB-IP-3 0.0400	0.0388	0.0049	1.592	40.05	61.39	0.30
GB-IP-3 0.0400				28.45	63.95	0.70
0.010	2010.0	0.0050	1.586	30.00	62.41	0.42
_	0.1290	0.0054	1.586	29.27	63.01	0.73
						K
S-2 / AFR700B AF7-IP-1 0.0766 0.0	0.0766	9600.0	1.928	37.86	48.71	0.53
S-2 / AFR700B   AF7-IP-2   0.0745   0.0	0.0745	0.0093	1.933	38.07	48.58	-0.26
S-2 / AFR700B AF7-IP-3 0.0776 0.0	0.0776	0.0097	1.935	37.93	48.82	0.14
S-2 / AFR700B   AF7-IP-4   0.0770   0.0	0.0770	9600.0	1.934	37.96	48.77	0.19
S-2 / AFR700B AF7-IP-5 0.0764 0.0	0.0764	9600.0	1.938	37.71	49.07	0.10
S-2 / AFR700B AF7-QI-1 0.1141 0.00	0.1141	0.0095	1.929	38.23	48.47	0.23

Calculated using fiber densities shown

below

ρ AS4 fiber = 1.79 g/cc

р імт fiber = 1.78 g/cc

 $\rho_{\,6781\,\text{fiber}\,=\,}$  2.46 g/cc  $^2$  Calculated using fiber densities shown above and resin densities shown below

1.27 g/cc p 3501-6 resin =

1.28 g/cc

1.44 g/cc ρ 5250-4 resin =

P AFR700B resin =

Composite Panel Inspections TABLE 6

Material	Panel ID	Grind Down <sup>III</sup>	C-Scan	Tg (° C) <sup>[2]</sup>	Cross-Section
AS4 / 3501-6	GE-IP-1	SO	Figure 2	163.1	Figure 8
AS4 / 3501-6	GE-QI-2	Š	Figure 2	190.4	Figure 8
IM7 / 5250-4	GB-IP-1	Š	Figure 2	263.8	Figure 7
IM7 / 5250-4	GB-IP-2	OK (B)	Not Available	265.2	Figure 7
IM7 / 5250-4	GB-IP-3	š	Figure 2	262.3	Figure 7
IM7 / 5250-4	GB-QI-3	š	Figure 2	240.3	Figure 7
S-2 / AFR700B	AF7-IP-1	ð	Figure 1	406.9	Figure 6
S-2 / AFR700B	AF7-IP-2	š	Figure 1	417.5	Figure 6
S-2 / AFR700B	AF7-IP-3	š	Figure 1	419.1	Figure 6
S-2 / AFR700B	AF7-IP-4	š	Figure 1	414.8	Figure 6
S-2 / AFR700B	AF7-IP-5	š	Figure 1	418.7	Figure 6
S-2 / AFR700B	AF7-QI-1	Š	Figure 1	398.1	Figure 6

TI OK signifies that the panel's ply layup

was correct [2] Tg measured via Thermal Mechanical Analysis (TMA) [3] Cross-Sections showed that 1/2 of the panel (diagonally) was incorrect layup. The other diagonal half was correct.

**TABLE 7** 

Test Specimen Identifications

		Open				Thermal	Hardness/
		Hole	In-Plane	Sandwich	n-Plane Sandwich Galvanic Oxidative	Oxidative	Thermal/
Material	Panel ID	Comp	Shear	Corrosion	Corrosion Corrosion	Stability	Stability Cross-Sec
AS4 / 3501-6	GE-IP-1	N/A	127	N/A	N/A	N/A	19 and
							50
AS4 / 3501-6	GE-QI-2	127	N/A	N/A	N/A	N/A	A/A
IM7 / 5250-4	GB-IP-1	N/A	117 and	19	N/A	130 and	16
			5253	5		91102	3
IM7 / 5250-4	GB-IP-2	X X	1834 [1]	1018 <sup>[2]</sup>	N/A	3160[3]	712[3]
IM7 / 5250-4	GB-IP-3	X N	3551	1927	N/A	6190	1318
			and			and	and
			5455		,	103116	20
IM7 / 5250-4	GB-QI-3	130	N/A	N/A	16	N/A	A/N
S-2 / AFR700B	AF7-IP-1	ΑΝ —	1324	N/A	- VA	2140	610
S-2 / AFR700B	AF7-IP-2	A/N	2536	ΝA	A/N	4160	N/A
S-2 / AFR700B	AF7-IP-3	A/N	112	ΥX	Υ <sub>N</sub>	120	15
S-2 / AFR700B	AF7-IP-4	ΑX	4960	Υ×	N/A	81100	A/N
S-2 / AFR700B	AF7-IP-5	K/N	3748	A/N	A/N	6180	N/A
S-2 / AFR700B	AF7-QI-1	131	A/N	N/A	N/A	Z/A	N/A
11 Specimone 20 24	Chocumona 20 24 was to be a basis to be an among	400,000					

' Specimens 28-34 were not used due to incorrect

panel layup 2) Specimens 14-18 had an incorrect layup, but used anyway due

to nature of the test [3] Specimens 31-60 and 7-12 were not used due to incorrect panel layup

**TABLE 8**Summary of Weight Gain Results Due to Deicer Exposure

		AS4/3501-6	01-6			IM7/5	M7/5250-4			S-2/AF	S-2/AFR700B	
			Ope	Open-Hole			Ope	Open-Hole			Oper	Open-Hole
	In-Plan	In-Plane Shear	Comp	Compression	In-Plar	In-Plane Shear	Comp	Compression	In-Pla	In-Plane Shear	Comp	Compression
	Mean	Std Dev	Mean	Std Dev Mean Std Dev	Mean	Mean Std Dev	Mean	Mean Std Dev Mean Std Dev	Mean	Std Dev	Mean	Mean Std Dev
Deice Fluid	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
UCAR PM-5197	0.066	0.007			0.197	0.004	0.099	0.000			0.587	0.010
Cryotech E-36	0.070	0.010			0.184	0.011	0.107	0.007			0.943	90000
UREA	0.133	0.005			0.410	0.016	0.121	900.0			0.700	0.008
Safeway SF	0.132	0.008			0.447	0.017	0.118	0.002			0.811	900.0
Octagon RD1431 SA			0.058	0.002			0.138	0.015			0.552	0.005

Comparison of IPS Test Results with the Baseline, Liquid and Solid Controls Table 9

					Delcer Fluids	=Inids				New York	S	Solid Deicers	íS
				(a (X))				Octagon				•	Octagon
Temp	Material	Strain	Baseline Control	UCAH PM-5197	Cryotech E-36	UREA	Sateway SF	HD1431 SA			Solid Control	Sateway SF	HD1431 SA
HT	AS4/	5% Axial	Dried	L:(+)	(±)	EBO	(#)	EBO	MOWER IT	I OLE	UREA	EBO	( <del>-</del> )
	3501-6			3.9 <sup>[2]</sup>	4.3	3.1	4.6	4:1:4				1.5	-4.4
FH	=	Ultimate	Dried	(±)	(+)	(+)	(±)	(+)	10.7545		UREA	EBO	(+)
				18.1	16.4	18.7	16.8	28.8				-1.6	8,4
R	/ /WI	5% Axial	Dried	CBO	( <del>+)</del>	EBO	EBO	EBO			UREA	(+)	EBO
	5250-4			-0.8	3.8	-0.5	2.6	-1.3	M. Chillian	(2)/47/		3.1	6.0-
FT	=	Ultimate	Dried	EB0	(+)	( <del>+</del> )	(+)	(+)			UREA	EBO	(+)
				-1.2	4:11	8.4	10.9	28.3				2.3	18.4
350°F	=	5% Axial	Dried	EBO	(·)	(-)	( <del>-</del> )	·			UREA	EBO	( <del>-</del> )
				-3.8	5.7	-3.7	6.1-	-13.0				1.9	9.6-
350°F	=	Ultimate	Dried	OE3	EBO	EBO	OBB	EBO	(IIIC);NEC   C		UREA	EBO	EBO
				-8.3	0.9	-6.9	3.4	-1.5				11.1	5.9
RT	S-2/	5% Axial	Dried	083	( <del>.</del> )	(-)	( <del>-</del> )	(-)			UREA	(·)	(•)
	AFR700B			-2.8	-22.3	-8.5	-11.5	-32.5	\$ 1176.3 VC 1101c			-3.3	-26.3
RT	=	Ultimate	Deird	083	(-)	(+)	093	( <del>.</del> )			UREA	(·)	(÷)
				-2.2	10.0	1.8	0.5	-8.7	- W. Kost K. S. Z. E.			-1.2	-10.3
550°F	=	5% Axial	Dujed	083	(9)	( <del>.</del> )	(-)	(-)			UREA	(-)	(-)
				-4.3	-11.3	-5.3	-13.6	-27.8	W. 1881 88 1/41 -			-8.8	-23.8
550°F	=	Ultimate	Deird	EBO	<u> </u>	EBO	EBO	EBO			UREA		EBO
				-5.0	-12.4	-0,7	-2.1	1.0				-1.4	1.7
100	000												

Motes: EBO used to denote where error bars overlap

(+) The strength of this group is greater than the control group (-) The strength of this group is less than the control group change in the data mean from the control

**TABLE 10** 

Comparison of OHC Test Results with the Baseline, Liquid and Solid Controls

				Dairer Fluids	spliil				Jahrel &	<b>.</b>	Solid Deicers	S
				1	2000		Octagon					Octagon
Test		Baseline	UCAR C	Sryotech		Safeway	RD1431		CANNON COMPANY	Solid	Safeway	RD1431
Temp	Material	Control   P	PM-5197	E-36	UREA	SF	SA	403000	Soft.	Control	SF	SA
RT	AS4 /	Dried	EBOII	EBO	EBO	EBO	EBO	THE STATE OF	1000	UREA	EBO	EBO
	3501-6		0.4 <sup>[2]</sup>	-2.8	-:	-3.3	-3.5	713 Con 18 10	600		-2.3	-2.4
RT	/ /WI	Dried	(+)	(+)	EBO	EBO	(±)			UREA	OB3	OBB
	5250-4		7.4	3.8	4.1	3.9	3.1	WHICE WAR			-0.2	-1.0
RT	8-2/	Dried	) (÷)	(-)	(-)	( <del>-</del> )	(·)			UREA	Ξ	( <del>-</del> )
	AFR700B		-12.0	-38.0	-10.0	-23.0	-16.0		5,000		-15.0	-6.9

EB0 used to denote where error bars T Notes:

overlap (+) The strength of this group is greater than the control group (-) The strength of this group is less than the control group

[2] % change in the data mean from the control

**TABLE 11**Summary of Hardness, Tg, and Cross-Section Results

Material	Sample Number	Deice Compound	Hardness (Barcol)	Tg (°C)	Cross-Section
AS4 / 3501-6	GE-H-5	Dried	. 62	162.4	No Damage
AS4 / 3501-6	GE-H-3	UCAR PM-5197	80	163.8	No Damage
AS4 / 3501-6	GE-H-1	UREA	79	163.6	No Damage
AS4 / 3501-6	GE-H-4	Cryotech E-36	79	162.2	No Damage
AS4 / 3501-6	GE-H-2	Safeway SF	80	162.6	No Damage
AS4 / 3501-6	GE-H-50	Octagon RD1431 SA	78	162.3	No Damage
IM7 / 5250-4	GB-H-5	Dried	81	258.9	No Damage
IM7 / 5250-4	GB-H-3	UCAR PM-5197	81	260.0	No Damage
IM7 / 5250-4	GB-H-1	UREA	80	259.8	No Damage
IM7 / 5250-4	GB-H-4	Cryotech E-36	81	259.9	No Damage
IM7 / 5250-4	GB-H-2	Safeway SF	81	259.5	No Damage
IM7 / 5250-4	GB-H-50	Octagon RD1431 SA	79	261.2	No Damage
S-2 / AFR700B	AF7-H-2	Dried	89	399.1	No Damage
S-2 / AFR700B	AF7-H-5	UCAR PM-5197	69	398.4	No Damage
S-2 / AFR700B	AF7-H-7	UREA	69	388.7	No Damage
S-2 / AFR700B	AF7-H-6	Cryotech E-36	65	402.3	No Damage
S-2 / AFR700B	AF7-H-8	Safeway SF	69	402.9	No Damage
S-2 / AFR700B	AF7-H-1	Octagon RD1431 SA	68	398.4	No Damage

**TABLE 12** 

Summary of Results from Sandwich Corrosion Testing

			Aluminum	Composite
		Corrosion		
		Severity		
Materials	Deice Compound	Rating <sup>[1]</sup>	Notes	Notes
IM7 / 5250-4 and 2024-T3	Dried	N/A <sup>[2]</sup>		N/A
IM7 / 5250-4 and 2024-T3	UCAR PM-5917	_		Cracking in outer layer of resin
IM7 / 5250-4 and 2024-T3	15% UREA	-		Cracking in outer layer of resin
IM7 / 5250-4 and 2024-T3	Cryotech E-36	-		Cracking in outer layer of resin
IM7 / 5250-4 and 2024-T3	15% Safeway SF	-		Cracking in outer layer of resin
IM7 / 5250-4 and 2024-T3	15% Octagon RD1431 SA	-		Cracking in outer layer of resin

<sup>[1]</sup> ASTM F1110 Corrosion Severity Rating System 0 = No visible corrosion (No Corrosion)

1 = Very slight corrosion or discoloration (up to 5% of the surface area corroded)
2 = Slight Corrosion (5 to 10%)
3 = Moderate Corrosion (10 to 25%)
4 = Extensive Corrosion or pitting (25% or more)

lil Not Applicable

#### Appendix List

Appendix A - Individual Specimen In-Plane Shear Test Results

Appendix B - Representative Stress-Strain Curves for In-Plane Shear Specimens

Appendix C - Individual Specimen Results for Open-Hole Compression

Appendix D - Representative Open-Hole Compression Stress-Strain Curves

# APPENDIX A Individual Specimen In-Plane Shear Test Results

# Individual Specimen Test Results for AS4/3501-6, IPS Testing @ RT

Deicing Fluid	15% Urea · "	:	15% Safeway SF "	UCAR PM-5197 "	Cryotech E-36
Date Tested	10-Nov-97	76-vov-51	10-Nov-97 " 12-Nov-97	10-Nov-97 " 12-Nov-97	10-Nov-97 12-Nov-97
5% Strain Stren. (lbs)	12.11	12.58 12.38 0.24 1.92	12.59 12.54 12.54 12.56 0.03	12.53 12.26 12.65 12.48 0.20 1.62	12.62 12.59 12.39 12.53 0.13
5% Strain Load (lbs)	1035	1107	1102 1095 1090	1095 1075 1095	1125 1125 1110
Thickness Max. Load Ult. Stren. (in.) (lbs) (ksi)	14.73	15.17 15.03 0.27 1.79	15.33 13.86 15.18 14.79 0.81 5.48	15.12 14.71 15.02 14.95 0.22 1.45	14.52 14.94 14.73 14.73 0.21
Max. Load (lbs)	1258 1308	1335	1342 1210 1320	1322 1290 1300	1295 1335 1320
Thickness (in.)	0.0430	0.0441	0.0439 0.0438 0.0436	0.0438 0.0440 0.0434	0.0447 0.0448 0.0449
Width (in.)	0.9934	0.9980	0.9971 0.9967 0.9970	0.9978 0.9966 0.9969	0.9974 0.9974 0.9979
Condition of Test	FR FR	ж <del>Г</del>	RR	RT RT	TR TR
Specimen Number	GE-IP-1-1 GE-IP-1-2	GE-IP-1-3 Average Std. Dev. C.V. (%)	GE-IP-1-4 GE-IP-1-5 GE-IP-1-6 Average Std. Dev. C.V. (%)	GE-IP-1-7 GE-IP-1-8 GE-IP-1-9 Average Std. Dev. C.V. (%)	GE-IP-1-10 GE-IP-1-11 GE-IP-1-12 Average Std. Dev. C.V. (%)

Individual Specimen Test Results for AS4/3501-6, IPS Testing @ RT continued

	····		
non = =	none = =	UCAR PM-5197 Cryotech E-36 Water	15% Octagon RD1431 SA "
10-Nov-97 " 12-Nov-97	24-Nov-97 25-Nov-97 26-Nov-97	4-Mar-98 "	7 May 98 5 May 98 6 May 98
12.17 11.84 < 5% strain 12.01 0.24 1.96	12.048 12.181 12.165 12.13 0.07	12.86 12.44 12.87	11.765 11.868 11.874 11.84 0.06
1103 1065 failed @ <	1065 1070 1065		1074 1082 1046
13.93 12.23 11.83 12.66 1.12 8.81	12.13 13.32 13.88 13.11 0.89	15.10 14.18 15.59	15.66 16.41 16.82 16.30 0.59
1262 1100 1055	1072 1170 1215		1430 1496 1482
0.0454 0.0451 0.0447	0.0443 0.0440 0.0441	0.0426 0.0432 0.0420	0.0458 0.0457 0.0450
0.9979 0.9972 0.9978	0.9977 0.9982 0.9926	0.9949 0.9958 0.9925	0.9966 0.9975 0.9788
R R F F	RR	RT RT	RT RT
GE-IP-1-13 GE-IP-1-14 GE-IP-1-15 Average Std. Dev. C.V. (%)	GE-IP-1-16 GE-IP-1-17 GE-IP-1-18 Average Std. Dev. C.V. (%)	GE-IP-1-19 GE-IP-1-20 GE-IP-1-21	GE-IP-1-26 GE-IP-1-23 GE-IP-1-24 Average Std. Dev. C.V. (%)

Notes:

GE-IP-1-5 Possible specimen damage due to misloading in the test machine. GE-IP-1-1 Started with a Grip Pressure of 750 psi an increased to 950 psi during the test to prevent slippage, all other tests used 950 psi.

GE-IP-1-14 Site of Specimen Failure Noted Early in the Test.

GE-IP-1-15

Individual Specimen Results for IM7/5250-4, IPS Testing at Ambient and 350°F

Notes	Grip Failure		Grip Failure
Deicing Fluid	15% Urea "	15% Urea "	25-Nov-97 15% Sodium 26-Nov-97 Formate 24-Nov-97 "
Date Tested	25-Nov-97 26-Nov-97 24-Nov-97	25-Nov-97 25-Nov-97 26-Nov-97	25-Nov-97 26-Nov-97 24-Nov-97
% Wgt Gain due to Deicing Fluid Exposure	0.403 0.428 0.398	0.410 0.016 3.923 0.429 0.427	0.428 0.001 0.234 0.465 0.465 0.443 0.017
5% Strain Stren. (ksi)	12.87 12.52 12.59	12.66 0.18 1.45 12.13 11.91	11.88 0.27 2.25 13.01 13.15 13.00 13.05 0.09
Ult. Stren. (ksi)	15.01 15.67 15.48	15.39 0.34 2.21 14.91 14.43	13.82 1.49 10.74 15.84 16.08 15.32 15.75 0.39
Condition Ult. Stren. 5% Strain of Test (ksi) Stren. (ksi) (ksi)	RI RI	350°F 350°F 350°F	RI TR
Specimen Number	GB-IPS-1 GB-IPS-4 GB-IPS-6	Average Std. Dev. C.V. (%) GB-IPS-2 GB-IPS-3 GB-IPS-5	Average Std. Dev. C.V. (%) GB-IPS-7 GB-IPS-9 GB-IPS-12 Average Std. Dev. C.V. (%) C.V. (%)

Individual Specimen Results for IM7/5250-4 continued page 2 of 4

			Grip Failure <5% Strain <5% Strain <5% Strain	מוחס ב	Grip Failure	
_	25-Nov-97 15% Sodium 26-Nov-97 Formate 24-Nov-97 "		UCAR		Э Э Э Э	
_	25-Nov-97 26-Nov-97 24-Nov-97		24-Nov-97 26-Nov-97 25-Nov-97 10-Feb-98		24-Nov-97 26-Nov-97 25-Nov-97	
	0.450 0.457 0.470	0.459 0.010 2.211	0.198 0.201 0.193	0.197 0.004 2.048	0.173 0.195 0.183 0.184 0.011	5.997
	12.10 12.23 12.00	12.11 0.12 0.96	10.64 12.88 13.04 13.18	12.62 1.12 8.86	13.19 13.20 13.19 13.20 0.01	0.06
_	15.32 15.36 15.41	15.36 0.05 0.31	10.64 12.88 15.55 15.30	14.03 2.22 15.86	15.62 16.15 15.68 15.82 0.29	1.85
	350°F 350°F 350°F		R H H H H	=	RT RT	
_	GB-IPS-8 GB-IPS-10 GB-IPS-11	Average Std. Dev. C.V. (%)	GB-IPS-13 GB-IPS-16 GB-IPS-18 GB-IPS-43	Average Std. Dev. C.V. (%)	GB-IPS-19 GB-IPS-22 GB-IPS-36 Average Std. Dev.	C.V. (%)

Individual Specimen Results for IM7/5250-4 continued page 3 of 4	Grip Failure Grip Failure		Grip Failure		
-4 continue	Б-36 	non = =	none " "	Sodium Acetate	
r IM7/5250.	24-Nov-97 26-Nov-97 26-Nov-97	24-Nov-97 25-Nov-97 26-Nov-97	24-Nov-97 25-Nov-97 26-Nov-97		
Results fo	0.190 0.190 0.182	0.187 0.005 2.466			
secimen	12.19 12.17 12.08	12.15 0.06 0.49 12.26 12.81	12.72 0.42 3.29 12.25 12.34 12.43	12.34 0.09 0.73 12.95 13.22	13.02 0.18 1.37
vidual Sp	15.53 14.43 15.02	14.99 0.55 3.69 13.52 14.35	14.20 0.62 4.34 14.32 15.21	14.85 0.47 3.19 18.00 17.55	17.76 0.22 1.26
<u>Indi</u>	350°F 350°F 350°F	R TR	350°F 350°F 350°F	₩ = <b>=</b>	
	GB-IPS-20 GB-IPS-21 GB-IPS-35	Average Std. Dev. C.V. (%) GB-IPS-37 GB-IPS-38 GB-IPS-40	Average Std. Dev. C.V. (%) GB-IPS-39 GB-IPS-41 GB-IPS-42	Average Std. Dev. C.V. (%) GB-IPS-23 GB-IPS-24 GB-IPS-52	Average Std. Dev. C.V. (%)

Individual Specimen Results for IM7/5250-4 continued page 4 of 4

		<5% Strain <5% Strain	
Sodium Acetate		UCAR: : : :	
		24-Nov-97 26-Nov-97 25-Nov-97 10-Feb-98	
		0.206 0.208 0.188	0.201 0.011 5.489
10.67 11.34 10.83	10.94 0.35 3.19	11.07 10.84 12.43 12.55 12.48	11.87 0.85 7.12
15.82 15.14 13.92	14.96 0.96 6.45	11.07 10.84 14.89 15.53	13.62 2.45 18.02
350°F 350°F 350°F		350°F 350°F 350°F 350°F 350°F	
GB-IPS-39 GB-IPS-39 GB-IPS-39	Average Std. Dev. C.V. (%)	GB-IPS-14 GB-IPS-15 GB-IPS-17 GB-IPS-45 GB-IPS-46	Average Std. Dev. C.V. (%)

#### S-2/AFR700B In-Plane Shear Specimen Results

Specimen	Condition	Ultimate	5%	Date	Deicing	
Number	of Test	Strength (ksi)	Strain Strength (ksi)	Tested	Fluid	Notes
AF7-IPS-37 AF7-IPS-38 AF7-IPS-39	RT RT RT	17.57 17.11 17.14	8.59 8.39 8.34		15% Octagon RD1431 S	A
Average Std. Dev. C.V. (%)		17.27 0.25 1.47	8.44 0.13 1.54			
AF7-IPS-1	RT	18.00	12.74	17-Feb-98	UCAR PM-5197	All Specimens Failed
AF7-IPS-2 AF7-IPS-3	RT RT	18.31 19.17	11.95 11.80	18-Feb-98 19-Feb-98		Near Specimen Identification Label
Average Std. Dev. C.V. (%)		18.49 0.61 3.29	12.16 0.51 4.18			
AF7-IPS-4 AF7-IPS-5 AF7-IPS-6	RT RT RT	17.35 16.69 16.98	9.93 9.49 9.75	17-Feb-98 18-Feb-98 19-Feb-98	u	1) 11
Average Std. Dev. C.V. (%)		17.01 0.33 1.96	9.72 0.22 2.23			
AF7-IPS-7 AF7-IPS-8 AF7-IPS-9	RT RT RT	18.95 19.21 18.89	11.23 10.97 11.02	17-Feb-98 18-Feb-98 19-Feb-98	н	n n
Average Std. Dev. C.V. (%)		19.01 0.17 0.88	11.07 0.14 1.29			
AF7-IPS-10 AF7-IPS-11 AF7-IPS-12	RT	19.28 19.18 19.30	11.34 11.47 11.55	17-Feb-98 18-Feb-98 19-Feb-98	n	11 11
Average Std. Dev. C.V. (%)		19.25 0.06 0.32	11.45 0.11 0.94			

S-2/AFR700B In-Plane Shear Specimen Results, continued page 2 of 3

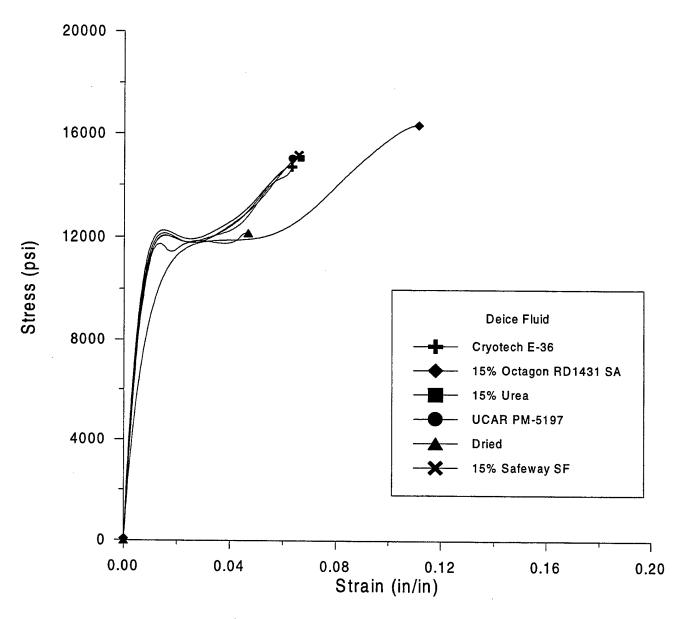
	5-2/AF	K/OOR	In-Plane	Shear	Specimen	Results, continue	ed page 2 of 3	
	455.00							ĺ
	AF7-IPS-13	RT	19.10	12.71	17-Feb-98		п	
	AF7-IPS-14	RT	18.69	12.61	18-Feb-98	1	"	l
	AF7-IPS-15	RT	18.94	12.21	19-Feb-98	lt	i ii	ı
								ı
	Average		18.91	12.51				ı
	Std. Dev.		0.21	0.27				ı
	C.V. (%)		1.10	2.14				
					į			ĺ
	AFR-IPS-47	550°F	12.59	6.57	27-May-98	15% Octagon RD1431	'SA	
	AFR-IPS-48	550°F	12.05	6.48	28-May-98	ı ı	1	
	AFR-IPS-60	550°F	12.15	6.17	28-May-98	n		
			;					İ
	Average		12.26	6.41				
	Std. Dev.		0.28	0.21				
	C.V. (%)		2.31	3.25			1	ı
	` ´			0.20				
	AF7-IPS-16	550°F	12.03	9.20	4-Mar-98	Dried	All Specimens Necked	
	AF7-IPS-17	550°F	11.96	8.61	4-Mar-98		" opecimens Neckeu	
	AF7-IPS-18	550°F	12.45	8.83	4-Mar-98		nt.	
			12.70	0.00	4-1VIAI-50	·		
	Average		12.14	8.88				
	Std. Dev.		0.26	0.30				
	C.V. (%)		2.18	3.34	;			
	3711 (70)		2.10	0.04				
	AF7-IPS-19	550°F	11.57	8.39	2-Mar-98	UCAR PM-5197	н	
	AF7-IPS-20	550°F	10.99	8.52	3-Mar-98		n'	
	AF7-IPS-21	550°F	12.03	8.61	4-Mar-98	11	tt	
				0.01	1 11101 00			
	Average		11.53	8.50				
	Std. Dev.		0.52	0.11				
	C.V. (%)		4.52	1.34				
	` ′							
	AF7-IPS-22	550°F	10.27	7.67	2-Mar-98	Cryotech E-36	II .	
	AF7-IPS-23	550°F	10.73	7.96	3-Mar-98		•	
	AF7-IPS-24	550°F	10.89	8.00	4-Mar-98	· n	lt .	
ĺ								
	Average		10.63	7.88				
	Std. Dev.		0.32	0.18				
	C.V. (%)		3.01	2.27				
	AF7-IPS-49	550°F	12.29	8.58	2-Mar-98	15% Urea	п	
	AF7-IPS-50	550°F	11.38	8.29	3-Mar-98	11	n	
	AF7-IPS-51	550°F	12.48	8.35	4-Mar-98	u	n	
	Average		12.05	8.41	i			
	Std. Dev.	1	0.59	0.15				
	C.V. (%)	İ	4.90	1.82				
•	•	•	•		1	1		

#### S-2/AFR700B In-Plane Shear Specimen Results, continued page 3 of 3

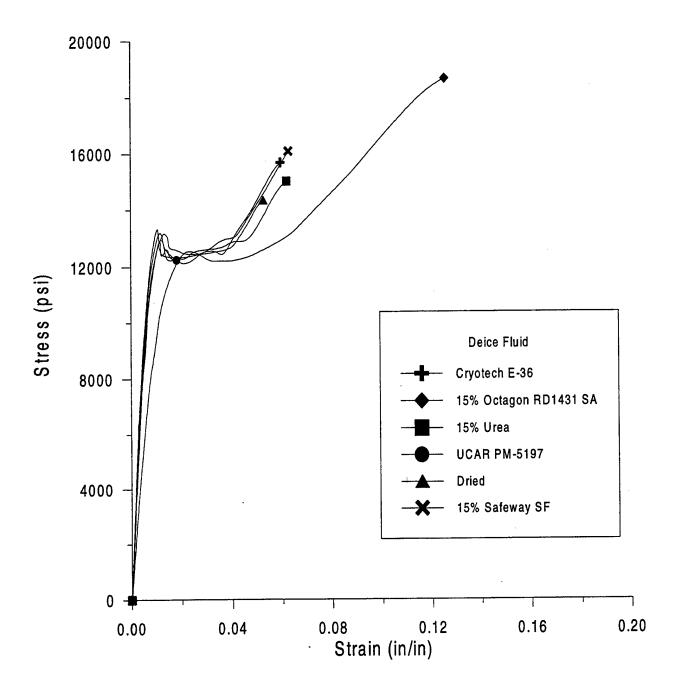
AF7-IPS-52 AF7-IPS-53 AF7-IPS-54	550°F	11.77 12.19 11.70	7.64 7.74 7.64	2-Mar-98 3-Mar-98 4-Mar-98	15% Safeway SF	11
Average Std. Dev. C.V. (%)		11.88 0.26 2.23	7.67 0.06 0.73			

Appendix B.

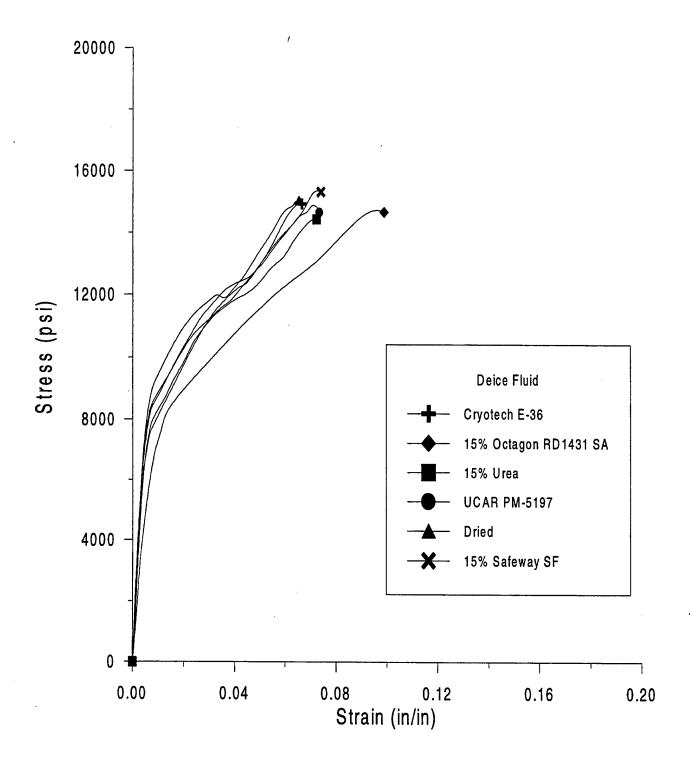
Representative stress-strain curves for in-plane shear specimens.



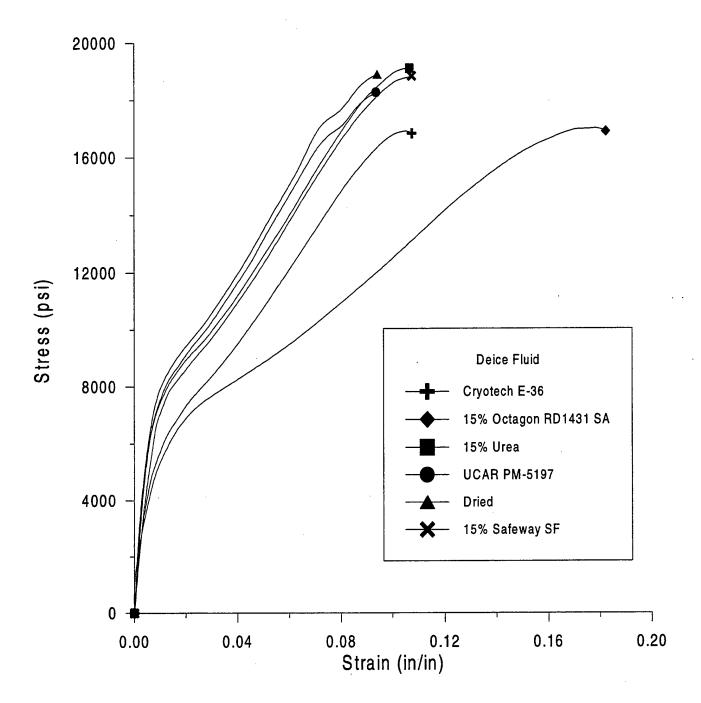
AS4/3501-6 (ambient temperature)



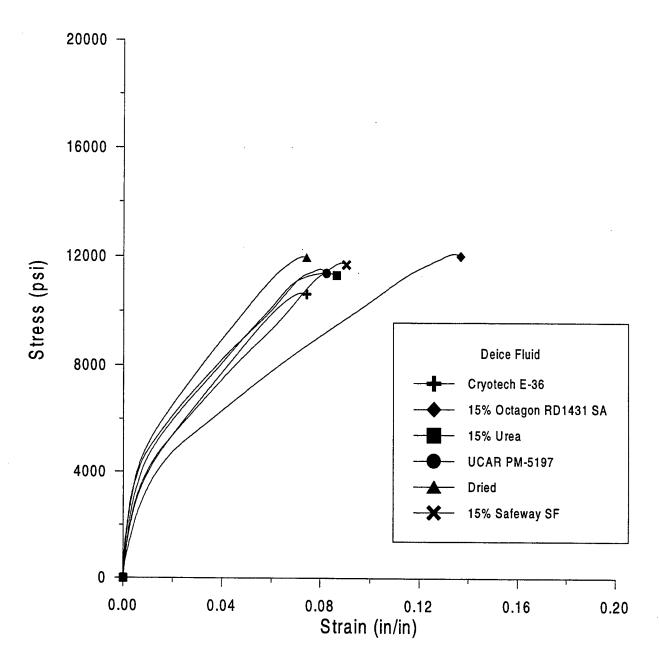
IM7/5250-4 (ambient temperature)



IM7/5250-4 (350°F)



S-2/AFR 700B (ambient temperature)



S-2/AFR 700B (550°F)

Appendix C Individual Specimen Test Results for Open-Hole Compression

#### **AS4/3501-6 Open-Hole Compression Individual Specimen Results**

Specimen	Conditio	Ult. Stren.	Deicing	
Number	n of Test	(ksi)	Fluid	Notes
GE-OHC-6 GE-OHC-17 GE-OHC-18		51.74 47.65 49.51	None "	Distilled Water Exposed
Average Std. Dev. C.V. (%)		49.63 2.05 4.13		
GE-OHC-4 GE-OHC-5 GE-OHC-7	RT RT RT	48.49 49.27 51.74	UCAR PM-5197 "	
Average Std. Dev. C.V. (%)		49.84 1.70 3.41		
GE-OHC-11 GE-OHC-12 GE-OHC-13	ľ	48.52 48.21 47.18	15% Safeway SF	
Average Std. Dev. C.V. (%)		47.97 0.70 1.45		
GE-OHC-14 GE-OHC-15 GE-OHC-16	RT	50.32 47.80 49.11	15% Urea "	Specimen Label Turned Darker and Appeared To Partially Dissolve
Average Std. Dev. C.V. (%)		49.08 1.26 2.57		
GE-OHC-8 GE-OHC-9 GE-OHC-10	RT RT RT	46.91 50.40 47.38	Cyrotech E-36	
Average Std. Dev. C.V. (%)		48.23 1.89 3.92		

#### AS4/3501-6 Open-Hole Compression Individual Specimen Results continued page 2 of 2

GE-OHC-1 GE-OHC-2 GE-OHC-3	RT RT RT	48.02 50.25 48.29	None "	Ambient "
Average Std. Dev. C.V. (%)		48.86 1.22 2.49		
GE-OHC-20 GE-OHC-21 GE-OHC-22	RT RT RT	46.77 47.34 52.03	none "	Vacuum Oven Dried
Average Std. Dev. C.V. (%)		48.71 2.89 5.92		
GE-OHC-23 GE-OHC-24 GE-OHC-29	RT RT RT	46.87 45.83 50.95	Octagon RD1431 SA "	
Average Std. Dev. C.V. (%)		47.88 2.70 5.64		

#### IM7/5250-4 Open-Hole Compression Individual Specimen Results

Specimen Number	Condition of Test	Ultimate. Strength (ksi)	Deicing Fluid	Weight Gain (%)	Notes
GB-OHC-1 GB-OHC-2 GB-OHC-3	RT RT RT	51.36 48.52 48.77	Distilled Water "	0.115 0.120 0.122	
Average Std. Dev. C.V. (%)		49.55 1.57 3.2		0.119 0.004 3.1	
GB-OHC-4 GB-OHC-5 GB-OHC-6	RT RT RT	51.20 49.46 51.64	UCAR PM-5197 "	error 0.099 0.099	
Average Std. Dev. C.V. (%)		50.77 1.15 2.3		0.099 0.000 0.1	
GB-OHC-7 GB-OHC-8 GB-OHC-9	RT RT RT	49.21 50.87 47.21	15% Safeway SF	0.117 0.121 0.117	
Average Std. Dev. C.V. (%)		49.09 1.83 3.7		0.118 0.002 1.6	
GB-OHC-10 GB-OHC-11 GB-OHC-12	RT RT RT	48.69 48.71 49.69	Cryotech E-36	0.100 0.110 0.112	Specimen Label Turned Darker and Appeared To Partially Dissolve
Average Std. Dev. C.V. (%)		49.03 0.57 1.2		0.107 0.007 6.2	
GB-OHC-13 GB-OHC-14 GB-OHC-15	RT RT RT	51.25 45.83 50.51	15% Urea "	0.114 0.127 0.122	
Average Std. Dev. C.V. (%)		49.19 2.94 6.0		0.121 0.006 5.2	

#### IM7/5250-4 Open-Hole Compression Individual Specimen Results continued page 2 of 2

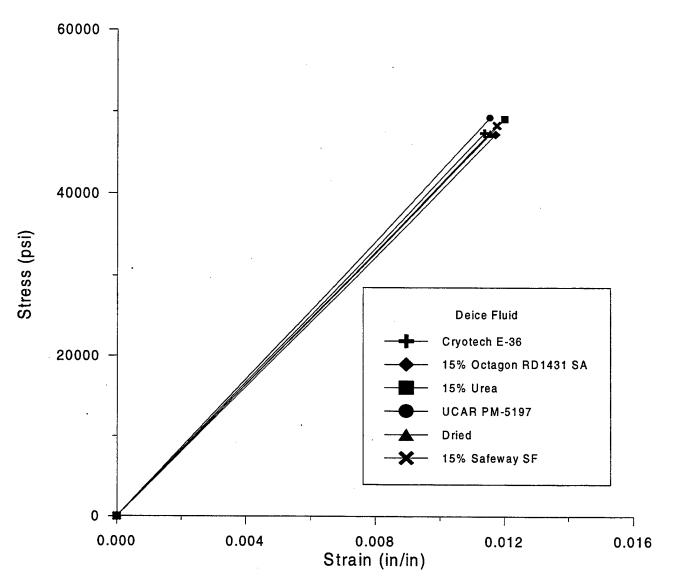
GB-OHC-16 GB-OHC-17 GB-OHC-18	RT RT RT	48.74 49.40 48.03	15% Octagon RD1431 SA	0.155 0.126 0.132	Ambient for 1 week
Average Std. Dev. C.V. (%)		48.72 0.68 1.4		0.138 0.015 11.1	
GB-OHC-19 GB-OHC-20 GB-OHC-21		47.56 46.89 47.27	none "		Vacuum Oven Dried
Average Std. Dev. C.V. (%)		47.24 0.33 0.71			

#### S-2/AFR700B Open-Hole Compression Individual Specimen Results

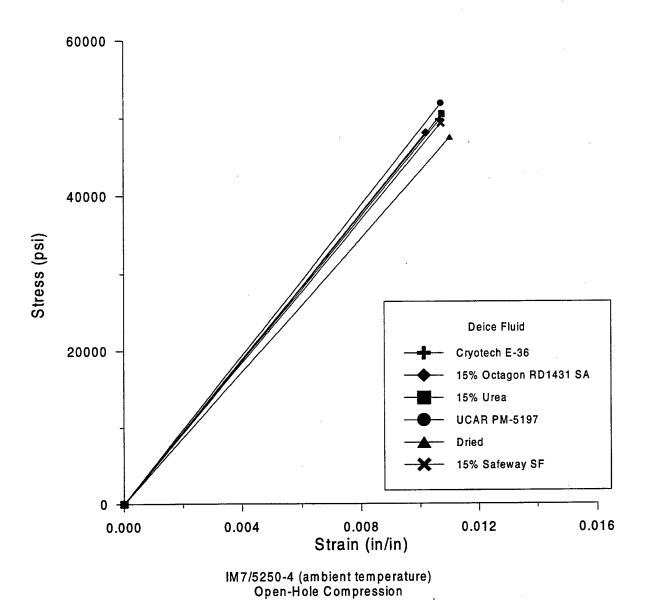
Specimen Number	Condition of Test	Ult. Stren. (ksi)	Deicing Fluid	Notes
AF7-OHC-1 AF7-OHC-2 AF7-OHC-3	RT RT RT	33.12 32.05 32.24	UCAR PM-5197 "	
Average Std. Dev. C.V. (%)		32.47 0.57 1.76		
AF7-OHC-4 AF7-OHC-5 AF7-OHC-6	RT RT RT	22.04 23.47 22.92	Cryotech E-36	
Average Std. Dev. C.V. (%)		22.81 0.72 3.16		
AF7-OHC-7 AF7-OHC-8 AF7-OHC-9	RT RT RT	32.64 33.34 33.60	15% Urea "	
Average Std. Dev. C.V. (%)		33.19 0.50 1.50		
AF7-OHC-10 AF7-OHC-11 AF7-OHC-12	RT RT RT	28.39 28.03 28.69	15% Safeway SF	
Average Std. Dev. C.V. (%)		28.37 0.33 1.15		
AF7-OHC-16 AF7-OHC-17 AF7-OHC-18		36.07 38.30 36.35	Dried "	
Average Std. Dev. C.V. (%)		36.91 1.21 3.29		% Wgt. Gain
AF7-OHC-13 AF7-OHC-14 AF7-OHC-15		30.30 32.05 30.31	15% Octagon RD1431 SA	0.557 0.547 0.551
Average Std. Dev. C.V. (%)		30.89 1.01 3.26		0.552 0.005 0.912

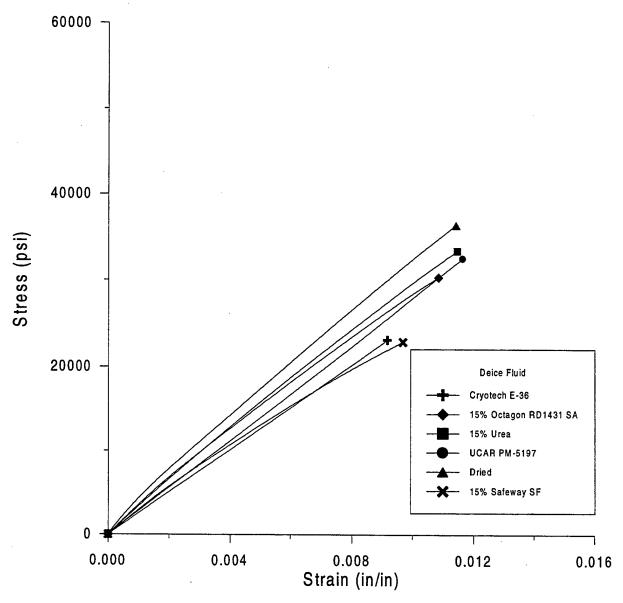
Appendix D.

Representative open-hole compression stress-strain curves.



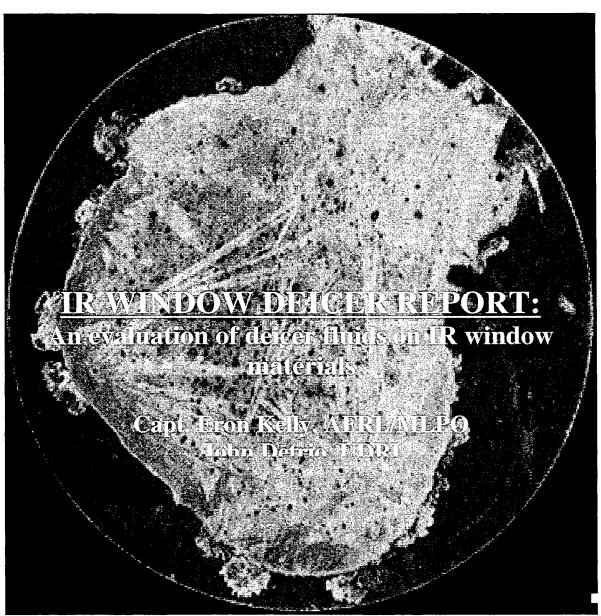
AS4/3501-6 (ambient temperature) Open-Hole Compression





S-2/AFR 700 B (ambient temperature) Open-Hole Compression

### APPENDIX 3 INFRARED WINDOWS



Stains left by sodium formate on a poly-silicon IR window sample.

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Table 2. IR window materials used during this study. Page 4

Table 3. Summary of transmission data. Page 5

### **PURPOSE:**

The purpose of this effort was to determine the effect of deicer fluids on standard Infrared (IR) window materials. The test was conducted to determine the effect of new environmentally friendly deicers on IR window materials.

# BACKGROUND:

This test was conducted using five deicer fluids and ten IR window material configurations. The deicers fluids used in this test can be found in Table 1, and the IR window materials tested are listed in Table 2. The pre-test and post-test measurements made on each sample included IR transmission spectra and digital macro photographs. Upon completion of the test, each sample was visually inspected for damage. The visual inspection and digital photographs coupled with the before and after transmission data were used to evaluate the affect of each deicer fluid on each window material.

well used to evaluate the affect of other services	
1. Control: Mil-D-83411 UCAR glycol/urea mix	
2. 15% solution of urea	
3. 15% solution of sodium formate	
4. Cryotech E-36 liquid potassium acetate	
5. Sodium acetate from Octagon	

Table 1. Deicer fluids used during this study.

IR Window Material	Anti-Reflection (AR) Coatings
Aluminum Oxynitride (AlON)	No coatings
Sapphire	No coatings
Zinc Selenide (ZnSe)	No coatings
Germanium (Ge)	No coatings
Poly-Crystalline Silicon (Poly-Si)	No coatings
Polymer Bond Layer	No coatings
Zinc Sulfide (ZnS)	Pilkington's Boron Phosphide Coating (side 1) Thorium Fluoride AR coating (side 2)
Zinc Sulfide (ZnS)	Raytheon's Rain Erosion Protection/Durable Anti-Reflection (REP/DAR) Coating (side 1) Yttria AR Coating (side 2)

Table 2. IR window materials used during this study.

The deicer test itself consisted of submerging each window material in a glass beaker full of deicer fluid. Within each 24 hour period during the five day test, the window materials would spend 4 hours immersed in deicer fluid and 20 hours out drying. After being removed from the deicer fluid for the last time, each window sample was washed with de-ionized water and dried with  $N_2$  gas.

### **FACTUAL DATA:**

The transmission spectra measured during this study were taken in the 8-11.5  $\mu m$  range for all samples except the AlON specimens. The AlON transmission spectra were measured in the 3-5  $\mu m$  region. Transmission measurements were not made on the sapphire samples because they did not have an optical polish, however there were no

visible stains or measured effect on any of the sapphire test samples. Also, the polymer bond layer did not suffer any ill effects from any of the deicers fluids. In Table 3 the transmission data is summarized for each window material and each deicer fluid.

		Transmission Loss in %				
Window Material	Pretest %	Control	Potassium Acetate	Urea	Sodium Formate	Sodium Acetate
AlON	88.73%	0%	0%	0%	0%	N/A
Ge	47.13%	0%	0%	0%	3.47%	0%
Poly-Si	47.43%	0%	0%	.91%*	4.28%	0%
ZnSe	71.50%	0%	.5%	1.03%	0%	0%
Polymer	50.99%	0%	1.11%	N/A	0%	0%
REP/DAR ZnS	70.32%	.53%	1.22%	0%	5.54%	2.33%
BP/ThF ZnS	66.71%	0%	0%	0%	1.74%	0%

Table 3. Summary of transmission data. \* Transmission loss may be due to scratches in sample generated during handling.

Pre and post test photographs were also taken of all the samples at each stage during the test. The pre-test photographs of several of the samples are shown in Figure 1. As the transmission data indicates, the sodium formate deicer stained many of the test samples. These stains were observed during visual inspection of each sample and they were recorded as digital photographs (Figure 2).

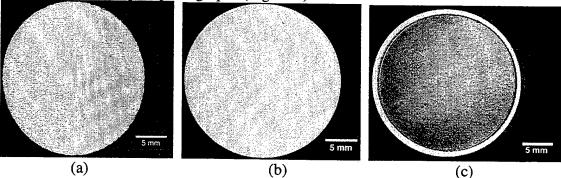


Figure 1. Pre-test photographs of (a) BP coated ZnS, (b) ZnSe, and (c), REP/DAR coated ZnS.

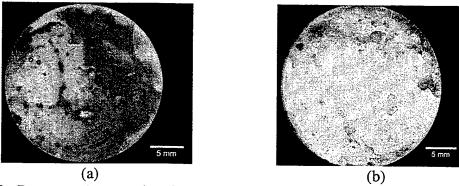


Figure 2. Post-test photographs of (a) Ge and (b) ZnSe that were stained by sodium formate.

### **DISCUSSION:**

After examining the data in Table 3, it is clear the sodium formate deicer has caused transmission loss in the Ge, Poly-Si and ZnS samples, while the other deicer fluids caused little or no change to the majority of IR window materials tested. The REP/DAR samples suffered some transmission loss because the back side AR coating was attacked by the two acetate deicers. This coating would never be exposed to the flight line environment, and thus would not come in contact with any deicer fluids. In this limited study only the sodium formate negatively affected many of the window materials. Close visual inspection and the digital photographs confirmed this phenomenon. The sodium formate stained many of the samples and the residue left behind is absorbing in the IR. These stains are easily identified by comparing before and after photographs of the stained samples (Figure 3).

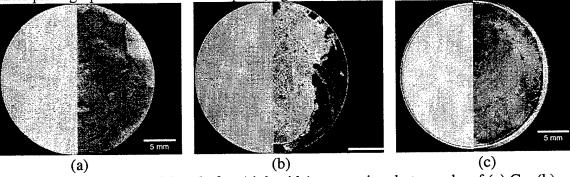


Figure 3. Before (left side) and after (right side) composite photographs of (a) Ge, (b) Poly-Si, and (c) REP/DAR ZnS IR window materials after being exposed to sodium formate.

An attempt was made to removed these stains first using water, then acetone and finally with alcohol. None of these basic solvents were able to remove the stains. When the transmission spectrum of each stained sample is closely examined, a small absorption band is found in the 8-11.5  $\mu$ m region. This band is due to the stain caused by the sodium formate deicer.

# **CONCLUSIONS:**

With the exception of the sodium formate, the deicer fluids tested in the study had little or no effect on the bulk of the IR window materials used in the Air Force today. The sodium formate deicer, however, stained the Ge, Poly-Si and ZnS IR window materials and left behind a residue that is absorbing in the IR. This residue did not come off when cleaned with common solvents like water, acetone and alcohol.

### **RECOMMENDATIONS:**

Sodium formate should not be used as a deicer fluid for Air Force systems that utilize exposed Ge, Poly-Si or ZnS IR windows. This deicer fluid will stain the window, reduce it's transmission and thus the performance of the system. If sodium formate must be used, it is recommended that an additional cleaning study be performed to determine whether or not a solvent exists that will remove the sodium formate stains from these window materials.

Advanced sensor windows will include complex coatings to meet optical and EMI requirements. Exposure to environmental hazards such as rain drop impacts, hail, dust or runway debris will damage the coatings and expose the constituent materials to chemical attack by deicing fluids and cleaning solvents. Any cleaning or deicing compounds considered for use on military aircraft that will come in contact with airborne sensor windows should be tested for compatibility with the window materials and coating systems.

This report was authored by Captain Eron Kelly of the Air Force Research Laboratory/Materials and Manufacturing Directorate, Electromagnetic Materials Development Section with assistance from Mr. John Detrio, The University of Dayton Research Institute.

# APPENDIX 4 ELASTOMERS/SEALANTS

# EFFECT OF RUNWAY DEICERS ON ELASTOMERIC MATERIALS

25 September 1998

# EVALUATION REPORT 4349IHRD/OC-AL

**REPORT NO. AFRL/MLS 98-152** 

# **AUTHOR(S)**

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ASC/YS

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# **AFRL/MLSA 98-152**

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# AFRL/MLSA 98-

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# **PURPOSE**

New environmental requirements dictate the use of alternate runway deicers. Compatibility of these new fluids with the materials found on the external surfaces and wheel wells of current aircraft must be tested. The purpose of this program was to determine the effects of five different deicers on elastomeric materials.

# **BACKGROUND**

Five deicers were used in this program including,

- a. CRYOTECH E36 LRD, manufactured by Cryotech Deicing Technology,
- b. SAFEWAY SF, manufactured by Clariant (Canada) Inc.,
- c. UREA, manufactured by Agricultural Minerals Corporation,
- d. UCAR PM-5197, manufactured by Union Carbide Corporation, and
- e. SODIUM ACETATE, manufactured by Octagone.

Seven elastomeric materials, including both bladders and sealants, were evaluated in the deicers. The bladder materials utilized were MIL-R-6855 Class I nitrile sheet and MIL-R-6855 Class II neoprene sheet. The sealant materials included the following:

- a. PR-1422 B-2, a MIL-S-8802F, Type I, polysulfide sealant,
- b. PS-870 B-2, a MIL-S-81733 corrosion inhibiting sealant,
- c. PR-1826 B-1/2, an AMS-3277 polythioether sealant,
- d. Q4-2817, an AMS-3375 fluorosilicone sealant, and
- e. PR-1750 B-2, an AMS-3276 polysulfide sealant.

# **TEST PLAN**

Tensile strength and percent elongation, 100% modulus, Shore A hardness, and percent volume swell were determined for both the nitrile and neoprene bladder materials. The sealants were evaluated for tensile strength and ultimate elongation, 100% and 300% modulus, Shore A hardness properties, percent volume swell, and peel strength properties on MIL-C-85285 gloss white topcoat. Tensile strength, ultimate elongation, 100% and 300% modulus, as well as peel strength properties were determined according to the requirements listed in the AS 5127 specification. Hardness properties were determined according to ASTM D2240.

The nitrile and neoprene materials were immersed in the deicers for 8 hours at room temperature then removed from the deicer for 16 hours at room temperature. This cycle was repeated 5 times. The remaining materials were immersed in the deicers for 4 hours at room temperature then removed from the deicer for 20 hours at room temperature. This cycle was repeated 5 times. PR-1750 B-2 was immersed in the deicers for both 4 hours and 8 hours at room temperature. The percent volume swell was measured for all materials after being immersed in deicer for 72 hours at room temperature.

# **FACTUAL DATA**

An increase in tensile strength and percent elongation was noted for the MIL-R-6855 Class I nitrile in all deicers, while no change in hardness properties or in 100% modulus properties was observed. The percent volume swells were less than 1% in CRYOTECH, SAFEWAY SF, and SODIUM ACETATE and less than 1% shrinkage was observed in UCAR and UREA. Table I lists the properties determined for the MIL-R-6855 Class I nitrile in all deicers.

The MIL-R-6855 Class II neoprene exhibited no loss of properties in any of the deicers. The tensile strength, percent elongation, 100% modulus, and Shore A hardness exhibited minimal change. The percent volume swell was less than 2% in all the deicers. Table II lists the properties determined for the MIL-R-6855 Class II neoprene in all deicers.

The PR-1422 B-2 exhibited an increase in tensile strength in all deicers with an 11% increase in CRYOTECH and an 18% increase in SODIUM ACETATE. The percent elongation decreased in all the deicers with the greatest loss occurring in UCAR. Both the 100% modulus and peel strength (100% cohesive failure) increased in all deicers. The original peel strength was 25 lbs with 100% cohesive failure. After immersion, the peel strengths ranged from 28 to 31 lbs with 100% cohesive failure. The Shore A hardness decreased in all deicers except CRYOTECH. A slight shrinkage of PR-1422 B-2 was observed in both CRYOTECH and UCAR, with slight swell in UREA and SAFEWAY. Table III lists all the properties determined for PR-1422 B-2 in all deicers.

A loss of tensile strength was observed for PS-870 in all deicers, except SODIUM ACETATE, with a 14 % loss in SAFEWAY SF. In addition a 10% loss of elongation was noted in the SAFEWAY SF deicer and a 22% loss in SODIUM ACETATE. The original peel strength was 53 lbs with 100% cohesive failure. After immersion the peel strengths ranged from 51 to 62 lbs with 100% cohesive failures. The 100% modulus increased in all deicers and the 300% modulus increased in CRYOTECH, UCAR, and SODIUM ACETATE. The PS 870 exhibited a slight shrinkage in CRYOTECH with volume swells ranging from 0.28% to 4% in the other deicers. Table IV lists all the properties determined for PS 870 in all deicers.

PR-1826 B-1/2 exhibited an increase in tensile strength and a loss of percent elongation in all deicers. The original peel strength was 65 lbs with 100% cohesive failure. After immersion the peel strengths ranged from 70 to 77 lbs with 100% cohesive failure. The percent volume swell varied from 0% to 2% in all deicers. Table V lists all the properties determined for PR-1826 B-1/2 in all deicers.

A loss of tensile strength and percent elongation was noted for Q4-2817 in all deicers. The peel panels were primed with DC-1200 prime coat. The coating on the control peel panels debonded during testing, however, after immersion in the deicers the coating did not debond during testing. The original peel strength was 10 lbs with failure occurring at the coating. The peel strength loads were 10 to 11 lbs after immersion in all deicers with 40 to 60% cohesive failure. The 100% modulus varied  $\pm 5$ % in all deicers, while the percent volume swell was 0 to 1% in all deicers. Table VI lists all the properties determined for QA-2817 in all deicers except UREA.

A 10% loss in tensile strength for the PR-1750 B-2, an AMS-3276 sealant was noted in CRYOTECH with a 13% loss of modulus noted in both CRYOTECH and UCAR. The percent elongation increased in all the deicers with the largest increase occurring in UREA. The original peel strength was 50 lbs. with 100% cohesive failure for PR-1750 B-2. After soaking, the peel strengths ranged from 3 to 6 lbs., with 100% adhesive failure in all deicers. The Shore A hardness increased slightly in all deicers and the percent volume swells ranged from 0.30 to 1.30 %. Table VII lists all the properties determined for PR-1750 B-2 in all deicers.

# **CONCLUSION(S)**

- 1. No significant changes were noted in the properties of either the nitrile or neoprene bladder materials due to immersion in any of the runway deicers.
- 2. A significant loss in peel strength was noted for PR-1750 B-2 sealant in all of the deicers.
- 3. A significant loss in % elongation was noted for PS-870 B-2 sealant in sodium acetate.

TABLE I

# Nitrile MIL-R-6855 Class I 5 days at RT

Test	Control	CRYOTECH	UCAR	UREA	SAFEWAY	SODIUM ACETATE
Tensile Strength(psi)	1978	1994	2031	2005	1960	2019
% Elongation	250	259	265	256	254	257
% Volume Swell	NA	0.60	- 0.58	- 0.20	0.24	0.74
Hardness	·			_		
(Shore A)	66	68	67	67	68	66
100% Modulus (psi)	791	775	778	792	779	789

# TABLE II

# Neoprene MIL-R-6855 Class II 5 days at RT

Test	Control	CRYOTECH	UCAR	UREA	SAFEWAY	SODIUM ACETATE
rest	Control					
Tensile Strength(psi)	971	965	954	950	961	965
% Elongation	167	169	169	168	169	171
Liongation						
% Volume Swell	NA	0.16	0.05	0.34	0.66	0.99
Hardness (Shore A)	65	67	68	67	67	62
			ļ			<del> </del>
100% Modulus (psi)	620	611	608	605	611	608

# TABLE III

# PR-1422 B2 MIL-S-8802,Type I 5 days at RT

Test	Control	CRYOTECH	UCAR	UREA	SAFEWAY	SODIUM ACETATE
Test	001111					
Tensile Strength(psi)	412	456	418	451	439	487
% Elongation	341	263	255	307	321	316
% Volume Swell	NA	- 1.10	- 0.30	0.60	0.60	2.43
Hardness (Shore A)	67	68	59	59	54	63
100% Modulus (psi)	254	328	306	303	288	318
300% Modulus (psi)	389	NA	406	436	428	470
Peel Strength(lbs)	25	31	31	30	28	30
% Cohesive	100	100	100	100	100	100

# TABLE IV

# PS-870 B2 MIL-S-81733 Corrosion Inhibiting 5 days at RT

Track	Control	СКУОТЕСН	UCAR	UREA	SAFEWAY	SODIUM ACETATE
Test	Control	CRICIZO				
Tensile Strength(psi)	460	435	440	407	394	466
% Elongation	630	575	580	601	565	493
% Volume Swell	NA	- 0.13	1.60	3.78	2.06	0.28
Hardness (Shore A)	47	44	47	43	46	51
100% Modulus	129	147	147	133	133	181
(psi)						
300% Modulus	251	275	275	245	244	340
(psi)		1				
Peel Strength(lbs)	53	54	57	61	56	51
3.4.3			100	100	100	96
% Cohesive	100	100	100	100	100	

# TABLE V

# PR-1826 B1/2 AMS-3277 Polythioether 5 days at RT

Test	Control	CRYOTECH	UCAR	UREA	SAFEWAY	SODIUM ACETATE
1 CSC	COMULOI					
Tensile Strength(psi)	443	516	447	469	465	485
% Elongation	455	347	310	350	382	339
% Volume Swell	NA	0.00	0.80	1.70	0.70	1.59
Hardness (Shore A)	48	54	52	52	51	55
100% Modulus (psi)	199	237	220	228	236	257
300% Modulus (psi)	366	489	452	446	421	465
Peel Strength(lbs)	65	72	70	72	77	75
% Cohesive	100	100	100	100	100	100

# TABLE VI

# Q4-2817 AMS-3375 Fluorosilicone 5 days at RT

Test	Control	СКУОТЕСН	UCAR	UREA	SAFEWAY	SODIUM ACETATE
Test	Control	02.13				
Tensile Strength(psi)	580	522	549	588	487	565
% Elongation	248	210	244	246	202	248
% Volume Swell	NA	0.2	0.9	1.0	0.9	0.25
Hardness (Shore A)	49	48	46	50	54	51
100% Modulus (psi)	445	425	425	436	452	493
Peel Strength(lbs)	10	10	11	11	10	12
% Cohesive	Coating	40	45	65	55	80

# TABLE VII

# PR-1750 B2 AMS-3276 Polysulfide 5 days at RT

Test	Control	СКУОТЕСН	UCAR	UREA	SAFEWAY	SODIUM ACETATE
Test	Control	CRIOIECH	CCAR	UKEA	BALLWAI	ACEIAIE
Tensile	<u> </u>					
Strength(psi)	547	488	495	505	509	524
%	242	248	272	282	270	271
Elongation						
	*********		·			
% Volume						
Swell	NA	0.29	0.42	1.32	0.91	0.43
Hardness						.
(Shore A)	53	54	54	57	58	56
100%						
Modulus	294	257	256	264	272	281
(psi)						
Peel						
Strength(lbs)	50	5	6	8	3	6
l l	30		U			
% Cohesive	100	0	0	8	0	6
Peel						
Strength(lbs)				7		
2						
				·		
% Cohesive				0		

<sup>&</sup>lt;sup>1</sup>8 hours at RT <sup>2</sup>4 hours at RT

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**PUBLICATION REVIEW:** This report has been reviewed and approved.

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# APPENDIX 5 ELECTRONICS

# Runway Deicer Aircraft Materials Compatibility Study (Materials Evaluation)

18 August 1998

**Evaluation Report** (4349IHRD/APPS)

Report No. AFRL/MLSA 98-134

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### **EXECUTIVE SUMMARY**

As a minimum, the conductivity, immersion, bend, voltage withstand and wet arc track propagation resistance tests should be used to evaluate potential deicing fluids and their effects on aircraft wiring insulations.

Conductivity should be used to screen deicing fluid candidates. Conductivity limits need to be established through further testing. Based solely on this study the conductivity should be much less than 4.0 mS/cm, the conductivity of UCAR. Ideally, the conductivity should be closer to urea at 667  $\mu$ S/cm.

The current conductive deicing fluids should not be used near exposed wiring or other electrical/electronic circuits.

### **ACKNOWLEDGMENTS**

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# Runway Deicer Aircraft Materials Compatibility Study

# **PURPOSE**

Determine any adverse effects of environmentally friendly runway deicers on Air Force aircraft materials, specifically wiring with different insulations.

# **BACKGROUND**

The intent of this study was to determine any adverse effects of environmentally friendly, runway deicers on Air Force aircraft materials. In the process, new candidate deicer solutions would be characterized and compared to existing deicers. This would lead to the development of additional test procedures for the qualification of runway deicers. These test procedures would ultimately be incorporated into AMS specifications 1431, "Solid Deicing/Anti-Icing Compound" and 1435 "Generic, Deicing/Anti-Icing Fluid." The approach was to identify materials of concern; anti-ice and deice materials for evaluation; test methods and exposure levels; conduct testing using current deicers for baseline and identify compatibility issues related to new deicers. AFRL/MLSA was tasked to analyze and evaluate the impact of these deicers on typical aircraft wiring.

# **FACTUAL DATA**

For the compatibility study a collection of five tests was run. These included conductivity, immersion, bend, voltage withstand and wet arc track propagation. For these tests, insulated silver-plated copper conductor wiring with four different insulation types was selected. All wiring was 20 gauge, except for the cable that contained two 22 gauge wires. These were as follows:

Polyimide insulation (81381/11-20)

Teflon® insulation (22759/11-20)

Hybrid construction insulation (22759/86-20)

Cable insulated twisted pair (22759/32-22(2))-Topcoat single wall, topcoat shield, cross-linked jacket.

The five candidate deicing solutions were:

UCAR (urea/propylene glycol) supplied as a water-based solution. It was used as supplied.

Cryotech (potassium acetate) supplied as a water-based solution. It was used as supplied.

Octagon (sodium acetate) supplied in solid form.

It was dissolved in water in the concentration of 15grams/100ml of water (13 percent by weight).

Sodium formate - supplied in solid form. It was dissolved in water in the

concentration of 15 grams/100ml of water (13 percent by weight). Urea - supplied in solid form. It was dissolved in water in the concentration of 15 grams/100ml of water (13 percent by weight).

# **Conductivity Measurements**

The conductivity of each of the deicing solutions was determined using a DC-4 Myron L. Company digital conductivity meter. This is a multirange instrument, capable of measuring liquids with conductivities up to 200 millisiemens per centimeter. Conductivity is a measurement of the ability of the sample to carry an electrical current. It depends on the actual and relative concentration of ionic species present, their valance and mobility. The concentration of dissolved gases contributes to an increased conductivity. The higher the conductivity of the solution, the more likely it can promote shorting through leakage currents and corrosion especially of metallic surfaces. As standards for comparison purposes, this instrument was used to measure the distilled water available in the laboratory and a 3 percent by weight sodium chloride solution. The 3 percent sodium chloride solution was measured because it is the typical solution used in the wet arc track propagation resistance test. To prevent contamination from sample to sample, the meter's measurement vessel was rinsed between samples with hot tap water, followed by distilled water. This was repeated until the distilled water conductivity was reached, indicating the measurement cup was sufficiently clean to resume testing. Each test fluid was measured three times and the values averaged to obtain the conductivity.

The results are compiled in appendix A. Distilled water from the laboratory had a conductivity of 6.8  $\mu$ S/cm. The 3 percent sodium chloride solution measured 81 mS/cm. Urea, while 100 times more conductive than distilled water, was the least conductive of the deicing fluid candidates at 667  $\mu$ S/cm. UCAR was six times the conductivity of urea measuring 4.06 mS/cm. Sodium acetate (octagon) measured 124 mS/cm. Sodium formate measured 177 mS/cm. The Cryotech solution was the most conductive deicing fluid candidate tested. As received, it measured greater than 200 mS/cm. A 10 percent solution of this Cryotech measured 105 mS/cm.

### Immersion Test

The immersion test was conducted using Test Method 601, Paragraph 4.6.1, "Fluid Immersion," of Society of Automotive Engineers (SAE) Aerospace Standard 4373, "Test Methods for Insulated Electric Wire," as a guide. This test was performed to determine the effect of the deicing materials on the wire insulation materials listed above. Three 24 inch lengths of each wire type were obtained for testing each deicer fluid. An additional 24 inch length of each wire type was obtained for control purposes. The outside diameter of each wire was measured in three spots along its length using Mitutoyo digital readout calipers. The spots chosen were at the center of each wire, and spots five inches to each side of center. Outside diameter measurement of the Teflon®, polyimide and hybrid insulation wires (all were 20 gauge) was straightforward in technique. Measurement of the cable, however, presented a problem, as the cable did not present a circular cross section. It was composed of two 22 gauge wires twisted together surrounded by an outer wrap. The diameter varied greatly depending on where measurements were made around a fixed location along the length of the wire. It was found that fairly consistent

measurements could be obtained by measuring the cable across the thickest points along a length of approximately one inch in the caliper jaws.

Plastic fixtures were fabricated to hold the wires in the prerequisite two inch diameter bend specified by the test method. The fixtures and wires were placed in trays holding the test solutions (figure 1). The solutions were stirred in the trays several times each working day during the exposure period. The solutions were used at room temperature. It was decided to submerge the wires in the test solutions for a period of 140 hours (5 days plus 20 hours) in order that removal would be at a convenient time for measurements.

After the 140 hours of exposure to the five test solutions, the wires were removed, washed and dried at room temperature for one hour. Diameter measurements were again made in the same three locations on each wire sample. No differences in diameter were noted in any of the wires. Apparently no swelling of the insulation had occurred to this point. A visual examination revealed no obvious deleterious effects to the insulation from the 140 hour soak in any of the fluids.

The wires were again placed in the fixtures and submerged in the test solutions for another 140 hours of exposure (5 days plus 20 hours). The samples were again removed, washed, and dried for one hour at room temperature. A final set of three diameter measurements was made for each wire and the results compared to the other two sets of measurements. The polyimide, Teflon®, and cable constructions did not change significantly in thickness after exposure in any of the five test solutions. The hybrid construction, however, did show a measurable increase in all of the solutions except the sodium formate. The increase averaged 0.06mm for the urea test solution, 0.03mm for the Cryotech (potassium acetate), 0.02mm for the wire in the UCAR (urea/glycol) test solution, and 0.02mm for the wire in the octagon (sodium acetate) solution. These were computed by averaging the three initial measurements and the three final measurements for each wire and then computing the differences between the initial and final average measurements. This was done for each solution. Data are compiled in appendix B.

### Bend Test

Following immersion testing, the wiring samples were subjected to the bend test. The bend test was conducted using Test Method 714, Paragraph 4.7.14, of SAE Aerospace Standard 4373, "Test Methods for Insulated Electric Wire," as a guide. This test was performed to determine the sensitivity of the insulation to cracking after immersion in the deicer solutions. The test configuration is shown in figure 2. The 20 gauge wires were tested using a 1.27 inch diameter mandrel and 1.0 pound winding tension weight. The cable that consists of two 22 gauge wires was tested using a 1.60 inch diameter mandrel and a 2.0 pound winding tension weight. Visual inspection of the wiring insulation found no cracks in any of the wire specimens. There were three instances where the insulation was damaged due to the tying action to the test fixture (2 cable (UCAR wire 1, Cryotech wire 3) and one Teflon® (sodium formate wire 2)) (figure 3).

# Voltage Withstand Test

Following bend testing, the wiring samples were subjected to the voltage withstand test. The voltage withstand test was conducted using Test Method 510, Paragraph 4.5.10, of SAE Aerospace Standard 4373, "Test Methods for Insulated Electric Wire," as a guide. This document referenced ASTM D 3032 "Standard Test Methods for Hookup Wire Insulation," Paragraph 8. This test was performed on all the wire samples (60) which had undergone immersion and bend testing, and on a control sample from each wire insulation type. This test was performed to determine insulation integrity following the immersion and bend testing. The testing was performed on a Beckman AC dielectric breakdown tester (figure 4). The peak voltage level of 2200 VAC was specified by the individual wire type specification. Due to the test instrument limitations, the sensitivity level was set at 500 VAC as compared to the 1100 VAC called for in the ASTM document. This meant the sensitivity was twice the specified value.

The solution bath was a 5 percent sodium chloride and 0.10 percent Triton X-100 wetting agent solution. The wires were 24 inches in length. Each wire sample had one inch of both ends stripped and twisted together. The wire specimens were soaked in the solution bath for a minimum of four hours. The insulation resistance between the conductor and the water solution was measured at 500 VDC to detect gross flaws. A HP 4329A high resistance meter was used to make these measurements. Three wires (two cable and one Teflon®), each having resistances less than 1E6 ohms, failed this test. Optical examination of the insulation revealed damage caused by mechanical handling/stripping action. The voltage withstand test was conducted by applying the voltage between the twisted ends of the conductor and the grounded solution bath. The voltage was increased from zero to 2200 VAC at a rate of 500 V/s. The peak voltage was applied for one minute. None of the samples failed this test and the insulation resistance values compared to the controls' values (appendix C).

### Wet Arc Track Propagation Resistance Testing

Arc track propagation resistance testing was conducted by Raytheon Technical Services Company in Indianapolis, Indiana. The testing consisted of electrical testing of bundles of MIL-W-81381/11-20 type wire using the MIL-STD-2223 Method 3006 test for wet arc track resistance. Instead of using salt water solution to drip on the bundles, each of the five deicing fluids was used to evaluate the relative arc track activity between the different fluids. Polyimide insulated wiring is the only test wiring insulation known to arc track. Primarily for this reason and secondarily due to time constraints and cost the test was conducted using only the one insulation type wiring. The test procedure is included in appendix D. The instrument used was a Lectromec Wet and Dry Arc Track Resistance Test System, Model 113094-01, with the wet test module (figure 5). The test was conducted using 0.5 ohm resistance and repeated using 2 ohm resistance. For the 0.5 ohm run there were three runs per deicing solution. At least two runs per deicing solution were accomplished at 2 ohm resistance. Further testing was not possible due to the wire samples being consumed. After completing the wet arc track test, the five initially undamaged wires were subjected to a wet dielectric test. Data accumulated during testing are compiled in appendix E.

Results of the arc track propagation resistance tests indicate the arc track propagation of a specific wire may be affected by the type of fluid that contacts the wires to promote short circuiting. Of the five deicer fluids used for testing, several trends were determined. Generally speaking, the 2 ohm tests produced greater damage when compared to the 0.5 ohm test. Typically, the damage length was twice as long, fewer wires passed, and the time to event was much shorter. This is due to the greater influx of energy and greater current flow with the larger resistance value. The 13 percent urea solution promoted the least amount of arc tracking in the wires. In addition, the sample bundles exhibited the least extent of damage with no collaterally damaged wires when an endpoint was reached (figure 6). The 13 percent octagon solution, 13 percent sodium formate solution, and the Cryotech solution all behaved similarly (figures 7 through 12). These were also the more conductive solutions. They all promoted some amount of arc tracking, and the extent of bundle damage was similar. Although, the damage with the Cryotech was somewhat less on average than the other two solutions. The collateral damage was also similar, except the Cryotech solution had fewer collaterally damaged wires on the 0.5 ohm test. The time required for the polyimide insulated wire bundles to reach an endpoint was the longest for the urea solution. The wire bundles tested with Cryotech and octagon solutions reached endpoints very quickly by tripping the circuit breakers. The wire bundles tested with the sodium formate solution were much more variable, with an event occurring after 9 seconds in one test and after 23 minutes in another test. The UCAR solution also was highly variable in all aspects, including the length of time to reach an endpoint. Wire bundles tested with the UCAR solution went to the extremes of no collaterally damaged wires in one sample to all damaged wires in another. Several of the bundles tested with UCAR solution ran as long as the urea, but with greater extent of bundle damage. It tended to promote small arcing for extended periods of time without tripping the breakers. This led to the slow erosion of the adjacent wire insulations. On average, the UCAR showed the most collateral damage and the greatest length of damage along the bundles when compared to the other four solutions (figures 13 and 14). The reason may be the circuit breakers were not easily tripped and the test ran for a longer time. For comparison, polyimide wires tested with the standard 3 percent sodium chloride or ammonium chloride solutions tend to have quick and devastating endpoints.

Representative wet arc track propagation resistance tests were video-documented and the tape is included.

# **CONCLUSION(S)**

There did not appear to be any deleterious effects on the wire insulations from immersion in the deicing fluids. The only exception to this is the hybrid construction wire that showed some insignificant swelling in all the fluids except sodium formate.

Immersion in the deicing solutions did not appear to impact the electrical or mechanical characteristics addressed by this study of the wire insulations. All the wires passed the bend and voltage withstand test following immersion test.

There is a correlation between conductivity of the deicing fluid and its ability to pass the wet arc track propagation resistance test. The greater the conductivity the more likely the

solution will promote and support arc tracking. Except for Cryotech, the greater the conductivity of the solution the more devastating the wet arc tracking event. All the deicing fluids tested, except for urea, failed the wet arc track propagation resistance test.

# **RECOMMENDATION(S)**

As a minimum, the conductivity, immersion, bend, voltage withstand and wet arc track propagation resistance tests should be used to evaluate potential deicing fluids and their effects on aircraft wiring insulations.

Conductivity should be used to screen deicing fluid candidates. Conductivity limits need to be established through further testing. Based solely on this study the conductivity should be much less than 4.0 mS/cm, the conductivity of UCAR. Ideally, the conductivity should be closer to urea at 667  $\mu$ S/cm.

The current conductive deicing fluids should not be used near exposed wiring or other electrical/electronic circuits.

# **FIGURES**

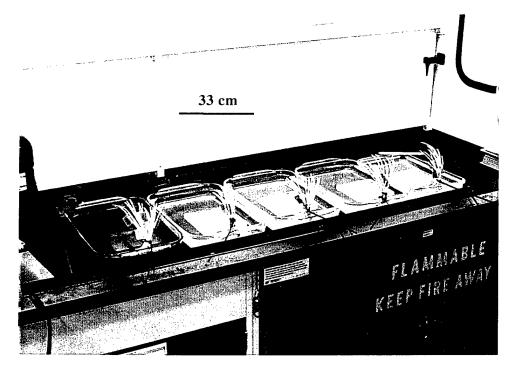


Figure 1. Immersion test setup. Shows the 12 wire samples undergoing immersion testing in each of the five deicing solutions.

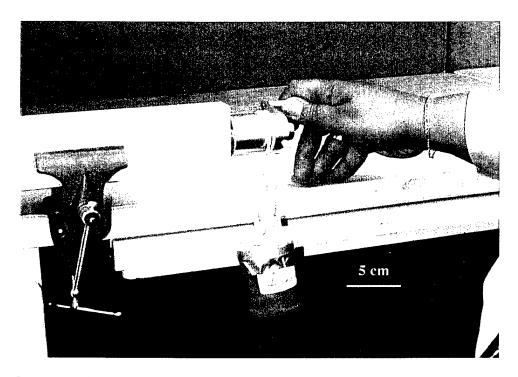


Figure 2. Bend test setup. Example illustrating the bend test of Teflon® insulation with one pound weight.

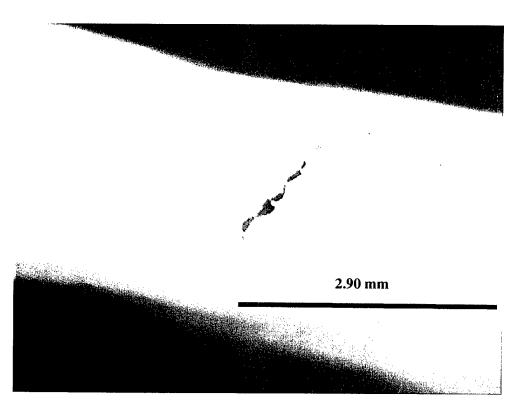


Figure 3. Typical damage caused by tying the cable wire to the bend test fixture. This cable had been soaked in Cryotech.

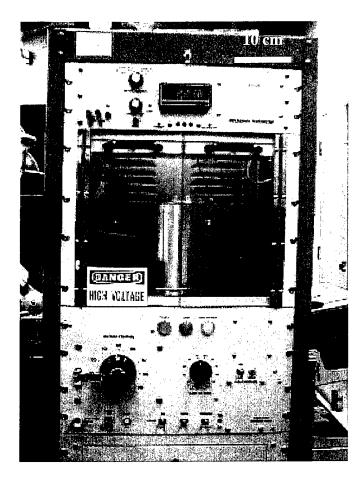


Figure 4. Beckman AC dielectric breakdown tester used in the voltage withstand testing. The can contains the salt/wetting solution and wire under test.

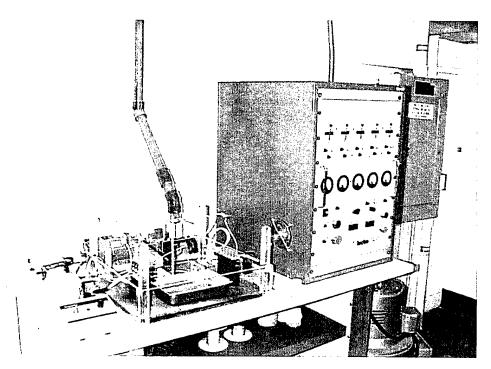


Figure 5. Wet arc track propagation resistance test system showing the peristaltic pump which delivers the solution, wire test connection and drip receptacle, and the Lectromec test system.

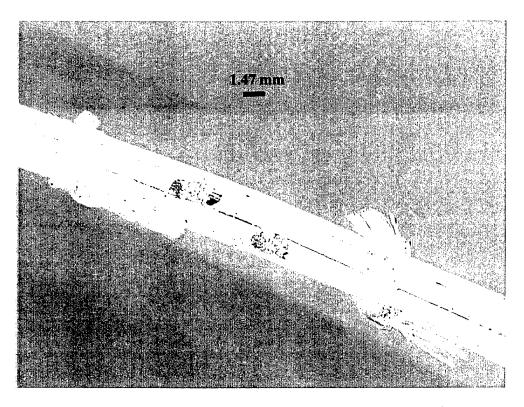


Figure 6. Example of polyimide insulated wiring which underwent wet arc track resistance testing (#27) with urea deicing solution and 2 ohms resistance. This specimen lasted eight hours without an event or any damage.

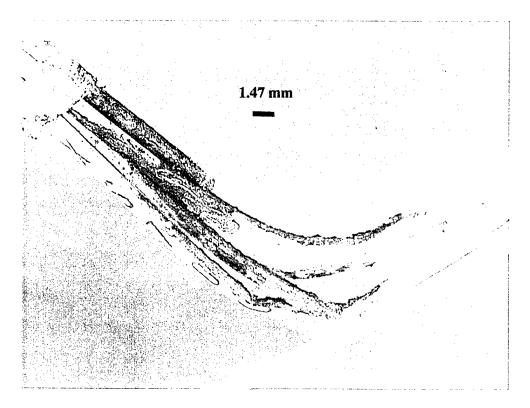


Figure 7. Example of polyimide insulated wiring which underwent wet arc track resistance testing (#14) with sodium formate deicing solution and 0.5 ohm resistance. This specimen went 4.9 hours until failure. Three wires failed and the maximum damage length was 0.863 inch. If the wires had remained horizontal, the time to failure would have been shorter.

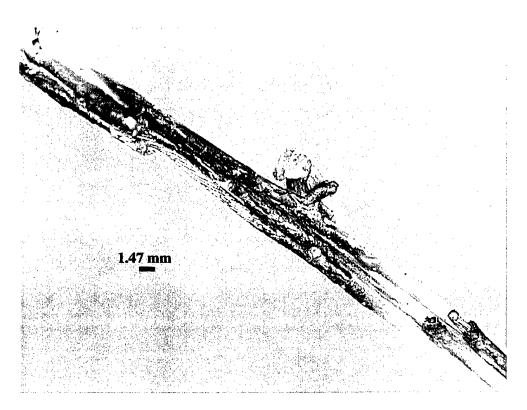


Figure 8. Example of polyimide insulated wiring which underwent wet arc track resistance testing (#26) with sodium formate deicing solution and 2.0 ohms resistance. This specimen went 48.9 seconds until failure. Two wires passed and the maximum damage length was 1.61 inches.

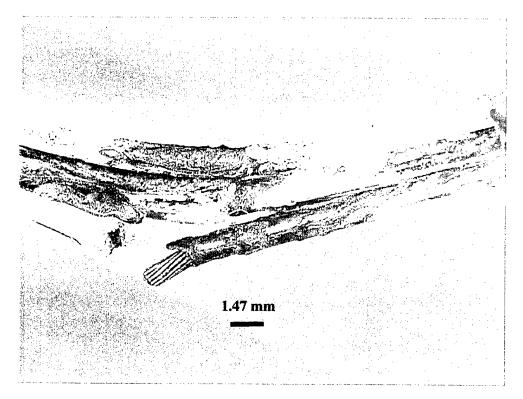


Figure 9. Example of polyimide insulated wiring which underwent wet arc track resistance testing (#3) with Cryotech deicing solution and 0.5 ohm resistance. This specimen went three minutes until failure. Five wires passed and the maximum damage length was 0.613 inch.

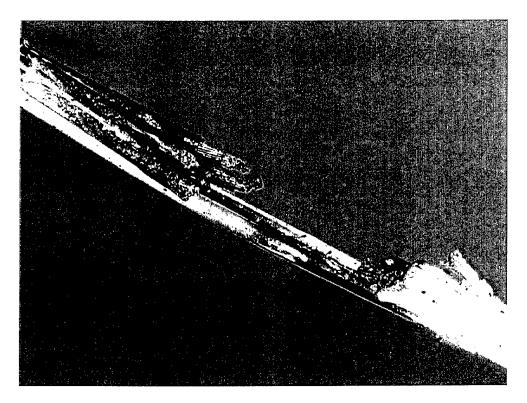


Figure 10. Example of polyimide insulated wiring which underwent wet arc track resistance testing (#18) with Cryotech deicing solution and 2.0 ohms resistance. This specimen went 6.6 seconds until failure. Two wires passed and the maximum damage length was 1.424 inches.

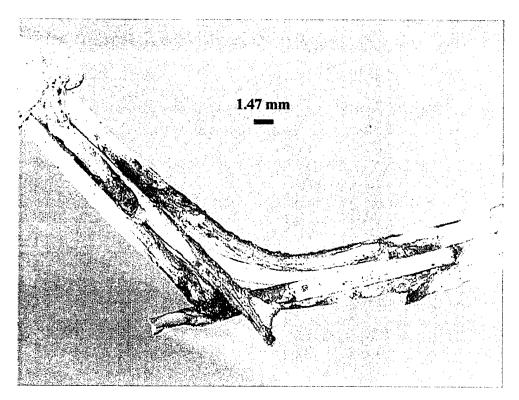


Figure 11. Example of polyimide insulated wiring which underwent wet arc track resistance testing (#11) with octagon deicing solution and 0.5 ohm resistance. This specimen went 20 seconds until failure. Three wires passed and the maximum damage length was 0.560 inch.

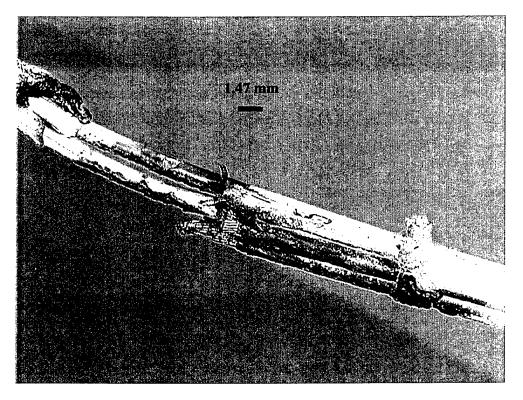


Figure 12. Example of polyimide insulated wiring which underwent wet arc track resistance testing (#17) with octagon deicing solution and 2.0 ohms resistance. This specimen went two minutes until failure. Two wires passed and the maximum damage length was 1.396 inches.

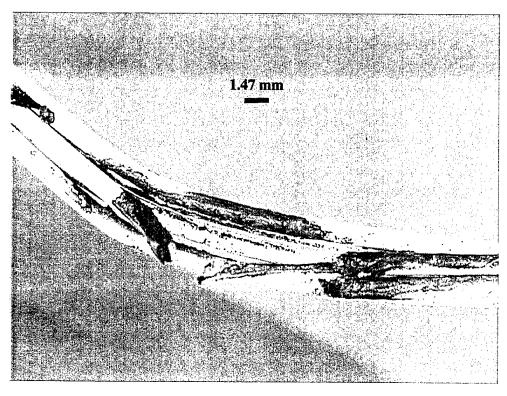


Figure 13. Example of polyimide insulated wiring which underwent wet arc track resistance testing (#6) with UCAR deicing solution and 0.5 ohm resistance. This specimen went 45.3 minutes to failure. Three of the wires passed and the maximum damage length was 0.795 inch.

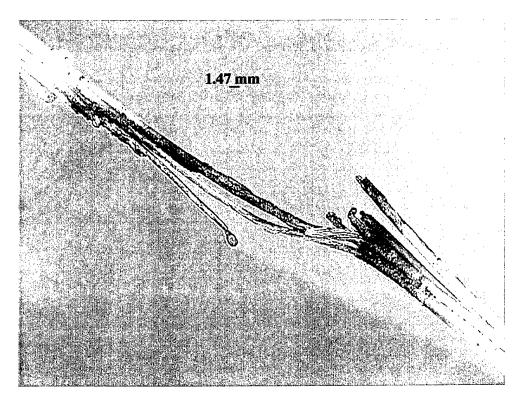


Figure 14. Example of polyimide insulated wiring which underwent wet arc track resistance testing (#22) with UCAR deicing solution and 2.0 ohms resistance. This specimen went four hours until failure. All of the wires failed and the maximum damage length was 2.67 inches.

**APPENDICES** 

Appendix A

### Conductivity Measurement Results

Test Solution	Measurements (S/cm)	Average Value (S/cm)
Distilled Water	6.8E-6	6.8E-6
Sodium Formate	177E-3	
	176E-3	177E-3
	177E-3	
Sodium Acetate	126E-3	
(Octagon)	123E-3	124E-3
	124E-3	
Urea	669E-6	
	682E-6	667E-6
	680E-6	
Cryotech (10% of as	1045.2	
received) (Potassium Acetate)	124E-3 100E-3	104E-3
(Fotassium Acetate)	91E-3	104E-3
3% Sodium Chloride	79E-3	017.0
	81E-3 82E-3	81E-3

Octagon (Sodium acetate 15g/100mL H<sub>2</sub>O% by weight)

Wire Type	Measure Point Nominal Wire	Nominal M	/ire Diameter (mm)	ir (mm)	Diameter (	Diameter (mm) 140 Hours	ours	Diameter (	Diameter (mm) 280 Hours	ours	Change
		wire 1	wire 2	wire 3	wire 1	wire 2	wire 3	wire 1	wire 2	wire 3	
Polvimide	-	1.47	1.46	1.46	1.47	1.46	1.46	1.47	1.46	1.46	0.00
31381/11-20	2	1.46	1.45	1.47	1.46	1.48	1.46	1.46	1.46	1.46	0.01
	ဧ	1.46	1.45	1.46	1.46	1.45	1.47	1.46	1.45	1.47	0.00
Teflon	-	1.47	1.48	1.48	1.46	1.47	1.47	1.46	1.48	1.47	0.00
22759/11-20	2	1.45	1.48	1.47	1.45	1.46	1.47	1.44	1.47	1.47	0.00
	3	1.48	1.47	1.47	1.47	1.48	1.47	1.47	1.46	1.46	0.00
Hybrid		1.28	1.31	1.26	1.30	1.30	1.27	1.29	1.30	1.30	0.01
22759/86-20	2	1.29	1.26	1.26	1.30	1.28	1.30	1.28	1.30	1.31	0.02
	ı e	1.28	1.27	1.27	1.28	1.29	1.28	1.29	1.30	1.29	0.04
Cable	-	2.90	2.90	2.90	2.90	2.90	2.88	2.83	2.90	2.87	0.00
22759/32-22	2	2.90	2.90	2.89	2.87	2.89	2.87	2.88	2.86	2.90	0.00
	3	2.89	2.89	2.89	1.89	2.87	2.88	2.85	2.90	2.88	0.00

Sodium Formate (15g/100mL  $H_2O = 13\%$  by weight)

Wire Type	Measure Point Nominal Wire	Nominal W	lire Diameter (mm)	er (mm)	Diameter (	Diameter (mm) 140 Hours	ours	Diameter (	Diameter (mm) 280 Hours	ours	Change	
Į.		wire 1	wire 2	wire 3	wire 1	wire 2	wire 3	wire 1	wire 2	wire 3		
Polvimide	-	1.47	1.45	1.47	1.46	1.47	1.47	1.47	1.46	1.46	0.00	
31381/11-20	2	1.47	1.46	1.47	1.47	1.46	1.37	1.47	1.46	1.47	0.00	
	3	1.46	1.47	1.47	1.46	1.46	1.46	1.46	1.46	1.46	0.00	
Fellon	-	1.46	1.48	1.49	1.47	1.49	1.45	1.45	1.46	1.47	0.01	
22759/11-20	2	1.46	1.48	1.49	1.48	1.48	1.47	1.47	1.48	1.46	0.00	
	3	1.48	1.47	1.49	1.47	1.48	1.47	1.47	1.47	1.48	0.00	
Avbrid	-	1.26	1.26	1.26	1.27	1.26	1.29	1.27	1.28	1.30	0.05	
22759/86-20	2	1.26	1.28	1.28	1.28	1.29	1.29	1.29	1.29	1.29	0.01	
	3	1.27	1.30	1.28	1.30	1.30	1.29	1.29	1.29	1.28	0.02	
Sable	-	2.92	2.89	2.91	2.91	2.87	2.86	2.86	2.86	2.88	0.00	
22759/32-22	2	2.90	2.90	2.91	2.87	2.88	2.90	2.85	2.85	2.85	0.00	
	3	2.90	2.89	2.92	2.88	2.90	2.90	2.83	2.82	2.89	0.00	

Urea  $(15g/100mL H_2O = 13\% by weight)$ 

Polyimide 1 81381/11-20 2				חשוועום	Dialitate (IIIIII) 140 mouls	SINO	Diameter (min) zoo nouis	202 (11111	CIBO	Glange
1 2	wire 1	wire 2	wire 3	wire 1	wire 2	wire 3	wire 1	wire 2	wire 3	
2	1.46	1.46	1.47	1.45	1.47	1.47	1.45	1.47	1.46	0.00
	1.46	1.47	1.47	1.46	1.45	1.46	1.46	1.45	1.48	0.00
3	1.46	1.47	1.46	1.46	1.46	1.47	1,47	1.46	1.47	0.00
-	1.46	1.47	1.47	1.46	1.47	1.47	1.45	1.47	1.46	0.00
2	1.46	1.48	1.47	1.47	1.47	1.48	1.46	1.46	1.45	0.00
က	1.47	1.48	1.48	1.46	1.46	1.47	1.46	1.46	1.46	0.00
-	1.24	1.25	1.23	1.80	1.31	1.31	1.33	1.34	1.31	90.0
2	1.28	1.25	1.25	1.29	1.30	1.30	1.32	1.31	1.30	0.07
က	1.27	1.26	1.27	1.30	1.30	1.29	1.31	1.31	1.29	0.05
-	2.90	2.89	2.89	2.87	2.86	2.85	2.86	2.87	2.90	0.00
2	2.89	2.92	2.91	2.85	2.85	2.87	2.87	2.93	2.90	0.00
8	2.89	2.89	2.89	2.84	2.89	2.85	2.90	2.90	2.91	0.01

Cryotech (Potassium acetate) as received

Change		00.00	00.00	-	0.01	0.01	0.00	0.00	0.00	0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.00 0.00 0.00 0.00	0.00 0.	0.00 0.	0.00 0.
lours	wire 3	1.47	1.48	1.46		1 45	1.45	1.45	1.45	1.45	1.45 1.46 1.27 1.30	1.45 1.46 1.27 1.30 1.29	1.45 1.46 1.27 1.30 1.29	1.45 1.46 1.27 1.30 1.29 2.89	1.45 1.46 1.27 1.30 1.29 2.89
mm) 280 H	wire 2	1.46	1.46	1.46		1 49	1.48	1.49	1.49	1.48	1.49 1.48 1.29 1.30	1.49 1.48 1.29 1.30	1.48 1.48 1.29 1.30	1.49 1.48 1.29 1.30 2.91	1.48 1.29 1.29 1.27 2.91 2.92
Diameter (mm) 280 Hours	wire 1	1.47	1.47	1.47		1 45	1.45	1.45	1.45	1.45	1.45 1.48 1.30 1.30	1.45 1.47 1.30 1.30	1.45 1.47 1.30 1.30	1.45 1.48 1.30 1.30 2.86	1.45 1.47 1.30 1.30 1.30 2.86
ours	wire 3	1.47	1.46	1.46	4.47	3	1.45	1.45	1.45	1.45	1.45	1.45 1.48 1.27 1.29 1.27	1.45 1.48 1.27 1.29	1.45 1.45 1.29 1.29 1.27 2.90	1.45 1.48 1.27 1.29 1.27 2.90 2.89
Diameter (mm) 140 Hours	wire 2	1.47	1.46	1.46	1.47		1.48	1.48	1.48	1.48	1.48	1.48 1.48 1.30 1.26	1.48	1.48 1.30 1.31 1.26 2.89	1.48 1.30 1.31 1.26 2.89 2.90
Diameter (	wire 1	1.47	1.46	1.46	1.47	:	1.47	1.47	1.47	1.47	1.47	1.47 1.28 1.30 1.25	1.47 1.28 1.30 1.25	1.47 1.28 1.30 1.25 2.89	1.47 1.28 1.25 1.25 2.89 2.91
er (mm)	wire 3	1.48	1.46	1.46	1 47		1.47	1.47	1.48	1.48	1.47 1.48 1.29 1.25	1.48 1.29 1.25 1.24	1.48 1.29 1.25 1.24	1.48 1.29 1.25 1.25 2.91	1.48 1.29 1.25 1.24 2.91 2.91
'ire Diameter (mm)	wire 2	1.47	1.48	1.47	1 48	2	1.48	1.48	1.48	1.48	1.48	1.48 1.47 1.27 1.29	1.48 1.47 1.27 1.26	1.48 1.27 1.26 1.29 2.91	1.48 1.27 1.26 1.29 2.91 2.90
Nominal W	wire 1	1.47	1.48	1.47	1 47		1.48	1.48	1.48	1.48	1.48	1.48 1.26 1.28 1.26	1.48 1.26 1.26 1.26	1.48 1.26 1.26 1.28 2.92	1.48 1.26 1.28 1.26 2.92 2.92
Measure Point Nominal Wire		1	2	3	-		2	3	3 8	3 8 1	3 3 2 2	3 2 3	3 2 1 3	3 8 1 3 5 1	3 2 1 2 1 2 2
Wire Type		Polyimide	81381/11-20		Teflon	101101	22759/11-20	22759/11-20	22759/11-20	22759/11-20 Hybrid	22759/11-20 Hybrid 22759/86-20	22759/11-20 Pybrid 22759/86-20	22759/11-20 Hybrid 22759/86-20	22759/11-20 Hybrid 22759/86-20 Cable	22759/11-20 Hybrid 22759/86-20 Cable Cable

Runway Delcer Test Data-Wire insulation Immersion Test According to SAE Aerospace Standard 4373 Test Method 601

UCAR (Urea/glycol) as received

Wire Tyne	Measure Point Nominal Wire	Wominal W	/ire Diameter (mm)	r (mm)	Diameter (	Diameter (mm) 140 Hours	ours	Diameter (r	Diameter (mm) 280 Hours	ours	Change
naki oliv		wire 1		wire 3	wire 1	wire 2	wire 3	wire 1	wire 2	wire 3	
Polvimide	-	1.47	1.47	1.45	1.46	1.46	1.46	1.46	1.47	1.46	0.00
81381/11-20	2	1.45	1.47	1.46	1.46	1.46	1.46	1.46	1.46	1.48	0.00
		1.46	1.46	1.47	1.46	1.47	1.46	1.46	1.46	1.46	0.01
Teflon	-	1.47	1.46	1.47	1.45	1.45	1.46	1.46	1.45	1.46	0.00
22759/11-20	2	1.48	1.46	1.46	1.46	1.46	1.46	1.47	1.46	1.46	0.00
27 1 1 20 1 7 7		1.47	1.48	1.47	1.46	1.46	1.47	1.47	1.46	1.47	0.00
Hybrid	-	1.24	1.30	1.27	1.27	1.30	1.28	1.27	1.31	1.31	0.05
22759/86-20	٥	1 23	1.32	1.29	1.25	1.30	1.25	1.27	1.32	1.28	0.01
22/20/20 50		1.29	1.30	1.31	1.27	1.30	1.29	1.28	1.31	1.32	0.01
Cable	-	2.91	2.90	2.89	2.84	2.82	2.83	2.88	2.87	2.87	0.00
22759/32-22	2	2.89	2.90	2.90	2.89	2.87	2.83	2.87	2.88	2.83	0.00
		2.89	2.92	2.91	2.87	2.86	2.87	2.85	2.86	2.89	0.00

Appendix C

## Voltage Withstand Test

Insulation Resistance at 500 VDC between conductor and solution (1 minute stabilization)

## UCAR (Urea/glycol) as received

Voltage withstand Insulation Resistance	(ohms)	1x10 <sup>13</sup>	1x10 <sup>10</sup>	2x10 <sup>13</sup>	2x10 <sup>14</sup>	1x10 <sup>14</sup>	1x10 <sup>14</sup>	2x10 <sup>13</sup>	2x10 <sup>13</sup>	5x10 <sup>13</sup>	700 VAC (1.5 sec) Nick caused by fixture	1×10 <sup>13</sup>	1x10 <sup>10</sup>
Voltage withstand		Ь	Ь	Ь	Ь	Ь	Ь	Ь	d	Ь	700 VAC (1.5 sec)	Ь	Ь
Sample		1	2	8	ľ	7	3	1	2	3	1	2	3
Wire Type		Polyimide	81381/11-20		Teflon	22759/11-20		Hybrid	22759/86-20		Cable	22759/32-22	

# Octagon (Sodium acetate 15g/100mL H<sub>2</sub>O = 13% by weight)

Wire Type	Sample	Voltage withstand	Voltage withstand Insulation Resistance
			(smho)
Polyimide	<del>-</del>	С.	1x10 <sup>13</sup>
81381/11-20	2	Ь	2x10 <sup>13</sup>
	ε	d	1x10 <sup>8</sup>
Teflon	1	d	2x10 <sup>14</sup>
22759/11-20	7	d	4x10 <sup>13</sup>
	8	d	1x10 <sup>14</sup>
Hybrid		Ь	2x10 <sup>13</sup>
22759/86-20	2	d ·	2x10 <sup>13</sup>
	3	Ь	2x10 <sup>13</sup>
Cable	1	Ь	1x10 <sup>10</sup>
22759/32-22	2	Ь	5x10 <sup>10</sup>
	ဗ	Ъ	2x10 <sup>10</sup>

Urea (15g/100mL  $H_2O = 13\%$  by weight)

Voltage withstand Insulation Resistance	(ohms)	2x10 <sup>12</sup>	7x10 <sup>12</sup>	2x10 <sup>13</sup>	7x10 <sup>13</sup>	8x10 <sup>13</sup>	1×10 <sup>15</sup>		2x10 <sup>13</sup>	2x10 <sup>13</sup>	3x10 <sup>13</sup>	1x10 <sup>13</sup>	1x10 <sup>13</sup>	1x10 <sup>13</sup>
Voltage withstand		d	d	Ь	d	d	d	,	Ь	d	d	Ь	Ь	d
Sample		1	2	3	1	2	3		1	2	3	1	2	8
Wire Type		Polyimide	81381/11-20		Teflon	22759/11-20			Hybrid	22759/86-20		Cable	22759/32-22	

### Controls

Wire Type	Sample	Voltage Withstand	Voltage Withstand Insulation Resistance
			(ohms)
olyimide	1	d	8 x 10 <sup>12</sup>
31381/11-20			
eflon	1	Ь	2 x 10 <sup>14</sup>
2759/11-20			
lybrid	1	Ь	3 x 10 <sup>13</sup>
2759/86-20			
			•
Sable	1	Ъ	2 x 10 <sup>13</sup>
22759/32-22			

## Voltage Withstand Test

Sodium Formate (15g/100mL  $H_2O = 13\%$  by weight)

Insulation Resistance	(swyo)	6x10 <sup>12</sup>	1x10 <sup>13</sup>	4x10 <sup>12</sup>	2x10 <sup>14</sup>	Nick caused by fixture	1x10 <sup>14</sup>	2x10 <sup>13</sup>	2x10 <sup>13</sup>	2x10 <sup>13</sup>	3x10 <sup>12</sup>	2x10 <sup>12</sup>	5x10 <sup>12</sup>
Voltage withstand		<b>a</b>	<b>d</b>	<b>d</b> .	۵	480 VAC (1 sec)	Ъ	d	Ь	Ь	Ь	d	d
Sample		-	2	3	1	2	3	-	2	3	1	2	3
Wire Type		Polyimide	81381/11-20		Teflon	22759/11-20		Hybrid	22759/86-20		Cable	22759/32-22	

## Cryotech (Potassium acetate) as received

			,		·····		y=			,			
Voltage withstand Insulation Resistance	(smto)	1x10 <sup>13</sup>	1x10 <sup>13</sup>	8x10 <sup>12</sup>	4x10 <sup>13</sup>	7x10 <sup>13</sup>	2x10 <sup>14</sup>	2x10 <sup>13</sup>	1x10 <sup>9</sup>	2x10 <sup>13</sup>	8x10 <sup>12</sup>	9x10 <sup>12</sup>	Nick caused by fixture
Voltage withstand		Ф	ď	Ь	<b>с</b> .	Ь	ď	Д	d	d	Ь	d	0 VAC
Sample		-	2	3	1	2	3	+	2	3	1	2	3
Wire Type		Polyimide	81381/11-20		Teflon	22759/11-20		Hybrid	22759/86-20		Cable	22759/32-22	
stance						fixture							

### INTERCONNECTIONS ENGINEERING TEST LABORATORY

TEST PROCEDURE: #3006

TEST PROCEDURE TITLE: Wet Arc Ti

Wet Arc Track Resistance Test

SPECIFICATION(S)/REVISION: MIL-W-22759E

SLASH SHEETS: /80-/92

REQUIREMENT PARAGRAPH: As per Slash Sheet

Initial Qualification

1.0 PURPOSE- The wet arc-propagation resistance test for wire insulation provides an assessment of the ability of an insulation to prevent damage in an electrical arc environment. The test also evaluates the ability of the insulation to prevent further arc-propagation when the electrical arc is re-energized.

**2.0 TEST EQUIPMENT-** The Wet Arc Track Resistance Test is performed on the Lectromec Wet and Dry Arc Track Resistance Test System, model 113094-01, with the Wet Test Module, located along the south wall of Rm. 16, B/5000. This procedure is to be supplemented with the Lectromec Installation Operations Maintenance Manual.

### Materials needed for bundle preparation/\*testing:

- 1. \*Isopropyl alcohol.
- 2. \*Cloth.
- 3. Calipers.
- 4. Razor blades or razor knife.
- 5. Marking labels with felt tip marker.
- 6. Wire strippers.
- 7. \*Ohmmeter or continuity tester.
- 8. \*6-12" ruler.
- 9. Lacing Tape.
- 10. \*Allen wrenches.
- 11. \*Straight blade screwdriver.
- 12. \*Replacement micro-bore (drip feed) tubing.
- 13. \*Wire samples (15 bundles of 7 wires 17" length, 150 feet required, 200' preferred).
- 14. \*Very fine stiff wire to clear drip feed needle.
- 15. \*The Lectromec Installation Operations Maintenance Manual.
- 16. \*Flashlight.
- 17. \*(1)Liter (per 15 bundles) of Sodium chloride (NaCl) solution 3% by weight in distilled water (made by Mat'1 Lab).
- 18. \*Eye protection (safety glasses).

### See Operations Manual:

### Installing the Wet Test Module

- 1. Place the Wet Test Module into the module position area [Figure 2, Item K] such that the positioning standoffs [Figure 2, Item L] lie in the positioning holes [Figure 4, Item I]. The binding post stands [Figure 4, Item B] provide convenient handles for the Wet Test Module. The Wet Test Module is secured using the plastic, black colored, knurl headed, 10-32 screws
- 2. Mate the Wet Test Module circular connectors [Figure 4, Item J] with the Bench Top Test Unit circular connectors [Figure 2, Item H].
- 3. Connect the enclosure wire to the wet fixture base [Figure 4, Item K] using the screw on the back of the base as a post.

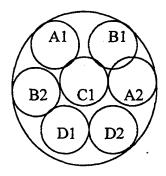
4. Push fit the long end of the electrolyte feed tube from the Wet Test Pump [Figure 2, Item A] into the needle connector (which is screwed into the needle) at the top of the Needle Rod. The other end of the electrolyte feed tube is placed in the Wet Test Fluid Reservoir [Figure 2, Item B], where the 3% Sodium Chloride solution (obtained from the chemistry lab) is to be added.

### 3.0 TEST SAMPLE - Bundle Preparation:

Cut the required wire lengths to be tested into approximate 50' bundles. Conduct a 2500 volt Wet Dielectric test on 100 percent of the wire in accordance with the Dielectric test procedure described in MIL-STD-2223, Method 3005, before the arc propagation resistance test is performed. Discard any failed sections of wire. Cut seven wire segments (17 inches) in length for each of the 15 bundles. (Note: Additional wires may be required to replace wires damaged in making the window cuts in A 1 and B1 wires.) Selecting two wires from each bundle (to become Al & Bl), mark the midpoint line of each with a fine felt tip marker and with a caliper, measure and mark an additional line 0.5-1 .0 mm (0.0197-0.0394 inch) from the first line. Using a razor blade knife, cut completely around (360 degrees) at the marks of the two wires, being cautious not to cut into the conductor. Make a lengthwise cut between these cut grooves and remove the insulation to expose the conductor. (Caution should be taken to avoid any damage to the conductor strands). Inspect each window cut under a microscope to assure strands are not cut and the window dimensions are correct.

Strip both ends of the seven wire segments. Clean the wires using a cloth with isopropyl alcohol. Five wire segments will be called "Active Wires" and two wire segments will be called "Passive Wires" for each bundle. Form the bundle by laying the seven segments straight and geometrically parallel. Assemble the wires to form six-around-one configuration shown below. The two pre-damaged wires should be placed in the Al and B 1 positions. Use Mil-T-43435 (type V) or equivalent lacing tapes to hold the test bundle together. Place labels denoting the circuit identification of the wire (Al, B1, etc.) near both ends of each wire, with care being taken to insure the wires are labeled correctly by using an ohmmeter or continuity tester. The two passive wires correspond to the D1 and D2 components shown. Check the bundle and wire orientation against the diagram below. Adjust the position of wires Al and B1 in the bundle to provide a longitudinal distance of 6.0 mm to 6.5 mm (0.2362 to 0.2560 inch) measured between the inner edges of each stripped window of the two exposed conductors.

### Bundle configuration ( side )



### Electrical connection (below)

Wire Identification	Power Supp	oly	Bundle Layer
Al	Phase A		Top
<b>B</b> 1	Phase B		Top
Cl	Phase C		Middle
A2	Phase A		Middle
B2	Phase B		Middle
D1	None	Passive	Lowest
D2	None	Passive	Lowest

**4.0 TEST PROCEDURE-** The Wet Arc Track Resistance Test is performed in accordance with test method 3006 of MIL-STD-2223.

- 1. Prepare a wire bundle for testing in accordance with MIL-STD-2223 method 3006 (see paragraph D2). Wire lengths of 17 inches are needed. If not previously performed, labels denoting the circuit identification of the wire (Al, A2, etc.) should be placed near both ends of each wire with care being taken to insure wires are labeled correctly. Clean the assembled bundle using a cloth and Isopropyl alcohol prior to installation in the test fixture.
- 2. Secure the wire bundle to the Wet Test Module using the wire bundle clamps [Figure 4, Item D].
- 3. Connect each of the five (5) active wires (A 1, A2, B 1, B2, and C2) to the correct binding post [Figure 3, Item C].

  Each wire must be connected with the correct terminal post by matching the terminal post labels and the labels (or stamped identification) denoting the circuit identification of the wire. The wires Al & B1 must be located on top with care taken to ensure that there is a longitudinal distance of 0.2362 to 0.2560 inch as measured between the inside edges of the stripped windows of the two conductors. The lacing tapes on the test wire bundle should be positioned such that the innermost ties are a nominal 1.5 inch apart to maintain wire bundle form. Slide the wire bundle into position such that the pre-stripped windows lie in the middle of the test drip zone.
- 4. Energize the system control power by pushing the Control Power Button on the Electronic Control Unit. When energized, the indicator light in the switch will be illuminated (green colored). Turn the Test selector switch to the "Wet" test position. The "Wet" indicator light (white) should be illuminated. (Note: There are two wet test switch positions. Both are equivalent. It does not matter which of these positions is selected.)
- 5. Locate the stainless steel pan under test sample to collect any excess drippage during test. Turn on the Wet Test Pump (in the forward direction) using the Pump Motor Controller [ Figure 2, Item N]. Ensure that the electrolyte drops are hitting the test wire bundle in accordance with MIL-STD-2223, Method-3006. (It has been found that a setting of around 2 on the Pump Motor Controller achieves the 8-10 drops per minute to the test specimen; however, the final rate must be checked due to variables of tubing and needle condition). If necessary, adjust the position of the needle by moving the Needle Rod [Figure 4, Item E] and Needle Rod Clamp [Figure 4, Item F]. Turn off the Wet Test Pump.
- 6. Place the cover on the protective case, closing the interlock switch. When the cover is properly positioned the Interlock Closed light (red) on the Electronic Control Unit will be illuminated.

### Executing: the Wet Arc Track Resistance Test:

- 1. Electronic Control Unit (ECU) Preparation (with Control Power "ON"):
  - a. Make sure that each of the five (5) circuit breakers (at the top of the front panel) are closed. All five (5) "Closed" lights (white) should be illuminated. Push the momentary hold continuity check on the ECU to assure all 5 active circuits are connected.
  - b. Select the circuit resistance for the test. This is accomplished by pointing the Circuit Resistance knob to the desired setting.

Perform the test on 3 bundles at each circuit resistance shown below;

Test Number	Circuit Resistance (ohms)
1-3	0.0
4-6	0.5
7-9	1.0
10-12	1.5
13-15	2.0

- c. Reset the Test Time to zero by pushing the reset bar on the top front of the Test Time clock.
- d. Reset the Elapsed Trip Time to zero by pushing the reset bar on the top front of the Elapsed Trip Time clock.

- 2. Make sure that the three 50 amp circuit breakers, mounted on the Motor-Generator, are closed then turn on the Motor-Generator by pressing the "ON" button on the motor starter.
- 3. Start exhaust fan by means of the wall switch located behind the ECU.
- 4. Start the flow of electrolyte solution by starting, from the Pump Motor Controller panel, the Wet Test Pump in the forward direction.
- 5. Energize the test power by pressing the Test Power "On" button on the Electronic Control Unit. The test time clock will start now. The Test Power light (red) should illuminate. The five (5) Continuity lights (green) should illuminate and the ammeters will read 1+/- 0.2 amps, in accordance with MIL-STD-2223 method 3006.
- 6.a. If the breaker for circuit Al or B1 trips -the Closed light (white) will turn "off" and the Open light (blue) will turn "on" for that circuit. The test power is automatically shut off. The Test Timer clock is stopped and Trip Time counter starts.
- b. If the breaker for circuit Cl, A2 or B2 trips, the TEST POWER REMAINS APPLIED TO THE TEST WIRE BUNDLE. After 180 seconds the test power will automatically be shut off and the Test Time clock stopped.
- c. For circuits that have lost continuity, the Continuity light (green) will not illuminate for that circuit. Test power will not be affected by loss of continuity during the Wet Arc Track Resistance Test.

### **5.0 TEST NOTES**

The following criteria are used to determine the test endpoint and test validity:

- a. If breakers Al or B 1 trip, watch the trip time indicator, after 3 minutes reset the tripped breaker and immediately reapply the test power. (Note: the flow of electrolyte solution continues to drip on the sample and if any extended time is noted between breaker trip and observation, it may be necessary to pad dry the sample prior to reset of test power). The test time will continue upon power reapplication. Continue the test for a total of eight hours (28.800 seconds) or until either phase Al or B1 circuit breaker has tripped twice. CAUTION: DO NOT RESET A CIRCUIT BREAKER THAT TRIPS TWICE. If any breaker trios (A 1 or B 1 for a 2nd time or A2. B2 or C 1 for the 1 st time), a test end point is reached.
- b. If a loss of continuity in any phase wire occurs (as may be indicated by an open circuit indicator), without tripping phase A 1 or B 1 circuit breaker, continue the test for a total of 8 hours or until a test endpoint is reached, which ever comes first.

When a test end point is reached, turn generator "OFF", switch pickup tube to fresh water source to purge salt water from tubing and needle (approx. 10 minutes), switch exhaust fan "OFF", switch Control Power "OFF", remove protective cover from sample test area, remove bundle from fixture, label the bundle and record test results (i.e. duration, breaker Al tripped at 18,600 secs, lost continuity on B2, no event in 8 hours). Use deionized or distilled water and a clean flux brush to gently remove excess salt deposits from the wire bundle and with a paper towel pat dry the wire bundle. (Note: Due to the nature of this test, it must be anticipated that only one test will be performed in the 8 hour potential duration. Thus it is advisable to prepare the next test sample for the next test day. Then all that is required is to switch water source, turn on control power, verify continuity, reset timers, set resistance, start generator and exhaust fan, press Test Power "ON", check amperage and verify drip rate.) Checks will be required every half hour to verify drip rate and assure reset of breakers, if necessary, to avoid extending the work day to achieve the 8 hour test as potentially required. Measure length of burn and perform wet dielectric test on all undamaged wires (excludes Al and Bl and any other wires that have exposed conductors). Perform the 1000 volt Wet dielectric procedure in accordance with MIL-STD-2223, Method 3005. See individual slash sheets for qualification criteria.

### Clean-up:

Use a paper towel and a cleaner safe for plexiglass on protective cover and warm water and sponge clean the entire wire holding fixture and the base of the wet arc fixture. Place needle connected to tubing into clean water source, to avoid drying and salt formation in needle until next day use.

### PREPARED BY

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PUBLICATION REVIEW: This report has been reviewed and approved.

MICHAEL F. HITCHCOCK, Branch Chief

Materials Integrity Branch Systems Support Division

Materials and Manufacturing Directorate

### APPENDIX 6 CARBON/CARBON BRAKES



### DEPARTMENT OF THE AIR FORCE HEADQUARTERS OGDEN AIR LOGISTICS CENTER (AFMC)HILL AIR FORCE BASE, UTAH

30 July 1998

MEMORANDUM FOR:

Lee Gulley AFRL/MLSC

2179 12th Street

Wright Patterson AFB, Ohio 45433

FROM:

Science and Engineering Laboratory

OO-ALC/TIELM

7278 4<sup>th</sup> Street Bldg 100 Bay D Hill AFB, Utah 84056-5205

SUBJECT: Deicer Testing on Carbon-Carbon Brakes

### I Background:

System program offices (SPO's) at Wright-Patterson AFB have brought to Wright Lab's attention material compatibility concerns with the newly acquired potassium acetate-based runway deicers. In response, a request was sent to all MAJCOMs to solicit their material concerns. Elimination of materials already covered in AMS 1431 or 1435 reduced the list. A final list of materials to undergo compatibility testing was proposed based on materials common across many aircraft systems and on materials believed to be the least resistant to degradation that could come in contact with the deicer chemicals. OO-ALC/TIELM had been tasked to look at carbon-carbon brake disk materials. The testing is to be accomplished with five different types of deicers.

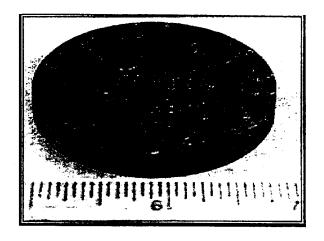
### 2. Objective:

The objective was to test the compatibility of the various types of carbon-carbon materials that are used on military aircraft with five deicing materials. Water was used as a baseline.

### 3. Materials:

a. The selection of carbon-carbon materials to be tested were determined by weapon systems, which include the B-2, C-17, C-5, F-15, F-16, and F-22. Each manufacturer has proprietary anti-oxidant coatings used for oxidation protection of their carbon-carbon brakes. Coupons with and without the coatings were tested.

Deicing Report, Page 1 of 5

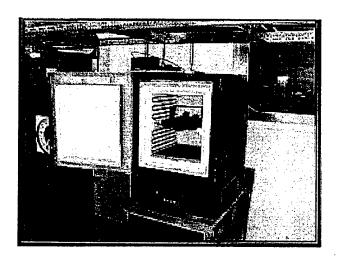


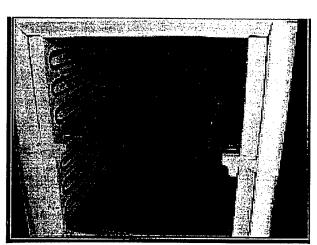
Photograph 1
Typical Carbon-carbon Disk

- b. Carbon-carbon brakes are fabricated either by laying up carbon-resin prepreg disks, chopped-cloth prepreg, or random fibers in carbon matrices. Two fabrications were represented in this testing matrix.
- c. The coupons were manufactured to approximately 2 inches in diameter and .25-.3 inch in thickness. ABS, Allied-Signal and BF Goodrich supplied the carbon-carbon materials (photo 1).
- d. The ABS coupons were taken from F-16 brakes that were in supply at Hill AFB. The part number for the brakes was 5007630-3 and the carbon-carbon was GY4452. The coupons were made in the tab and machined down to core material. There were 60 coupons, 30 non-coated and 30 coated with the anti-oxidant. The coating took place in the carbon-carbon shop at Hill AFB (LIOPWB). Coupons 2-15 were noted to have small cracks in the anti-oxidant coating after application and curing process.
- e. Allied-Signal provided coupons for four types of carbon-carbon: Carbenix 1000, 2000, 2110 and 4000. Each type of carbon-carbon had 30 coated and 30 non-coated coupons for a total of 240 coupons.
- f. BF Goodrich (BFG) provided coupons for two types of carbon-carbon, Super Carb and SPK. Two types of anti-oxidant coatings, M2 and M3, were used on the Super Carb material. Each configuration of carbon-carbon had 30 coupons supplied by BFG for a total of 150 coupons.
  - g. The deicing material to be tested was as follows: Deicer/Noun/Manufacturer
    - 1) Glycol mix/Ucar/Union Carbide.
    - 2) Potassium Acetate/E-36/Cryotech.
    - 3) Sodium Formate/Safeway/Old World Industries.
    - 4) Sodium Acetate/Octagon RD 1431 SA/Octagon Process INC.
    - 5) Urea/People's Moss Gin Co. INC.

### 4. Testing:

- a. Various tests were considered for evaluating the carbon-carbon materials, physical properties (hardness), mechanical strength (tensile/shear), simulation of performance (dynamometer) and oxidation resistance. The tests decided on were the oxidation resistance and hardness. The resistance to oxidation of a given material is measured from the weight loss per unit of time. The hardness test was done using a Shore D durometer.
- b. The coupons were cleaned and stored in plastic bags. They were handled with rubber gloves to avoid contamination. The oxidation test process was as follows:
  - Clean Coupons with Acetone in an Ultra-Sonic Bath (ABS carbon-carbon only, all other carbon-carbon coupons were cleaned at the manufacturer)
  - Initial Weighing and Hardness Testing
  - Contamination of Coupons with water, urea (solid), potassium acetate (liquid), sodium formate (solid), glycol mix (liquid), sodium acetate (solid). The coupons were immersed for 20 minutes. The solid deicers were mixed at 15 grams of solid to 100 ml of water, the liquids were used as is.
  - Dry Coupons at 110°F for 30 minutes.
  - Place in Oven at 1300°F for 4 hours (photos 2 and 3).
  - Cool with forced room air for 10 minutes.
  - Record weight after cooling.
  - Place in Oven at 1300°F for 4 hours.
  - Record weight and hardness after cooling.
  - Store in desiccator/plastic bags.

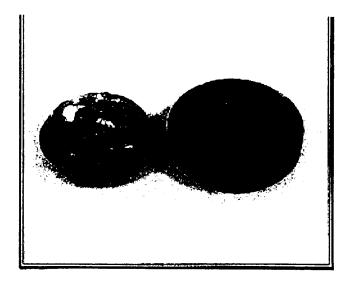




Photographs 2 and 3
Oven and Carbon-carbon Disk Test Future

### 5. Results/Discussion:

- a. All results can been examined in the attached charts and data sheets. Graphical representation of the *averages* of each type of carbon-carbon were compared with the specific contaminate. When viewing these graphs the % weight loss scale should be noted because the scales vary from graph to graph. Some data points were dropped in averaging of specific % carbon-carbon weights because the coupons were damaged. This damage was due to the end effects (excessive exposure) of the coupons, which were located at the end of the rows in the baking process.
- b. The charts and averages show significant differences between the % weight loss for the carbon-carbon materials that are coated compared to the carbon-carbon materials that are non-coated. The anti-oxidant coatings did provide protection for all the carbon-carbon materials from extensive oxidation after eight hours of exposure. The non-coated carbon-carbon materials showed a 3 to 5 times increase in oxidation after eight hours of exposure (photo 4). Both the water and urea generally had the least effect on the different carbon-carbon materials. The deicers with potassium and sodium had a slightly greater effect on the carbon-carbon materials than did the glycol mix. Potassium and sodium are considered as strong accelerators in the catalytic activity on the oxidation rate of carbon-carbon materials.



Photograph 4
Non-coated (left) and Coated( right)
Disks after Testing

- c. In carbon-carbon brakes the non-wear surfaces are the only areas coated with the antioxidant coatings. All wear surfaces are unprotected which constitutes the majority of the carbon-carbon brake. Coverage, and protection from anti-oxidant coatings is therefore minimal
- d. A loss in hardness for the majority of the coated and non-coated carbon-carbon materials was generally seen. The hardness on some coatings increased after baking. This is noted by the negative % loss values in the data. Some of the surfaces of the coupons were damaged and readings could not be taken. It could not be determined how

much the hardness losses effected the structural integrity of the carbon-carbon materials. Further mechanical tests would be needed to determine these properties and negative effects. Any loss in hardness should be considered detrimental to the integrity of the different carbon-carbon materials.

### 6. Conclusions:

- a. The deicing chemicals had a detrimental effect on the oxidation rate for the carbon-carbon materials at operating temperatures of 1300 degrees Fahrenheit.
- b. The water and the urea coupons were least effected by oxidation. The deicers with potassium and sodium had a greater effect followed closely by the glycol mix on the carbon-carbon materials.
- c. The anti-oxidant coatings provided good protection for the carbon-carbon materials.
- d. All of the non-coated carbon-carbon materials had loss in hardness. Any loss in hardness would be detrimental to the integrity of the carbon-carbon materials. Hardness increased on some coated carbon-carbon materials. Further study would be needed to determine the cause of this hardening effect.
- 7. If additional information is needed, please contact Steven Kmetzsch, DSN 777-9409, commercial 801-777-9409, e-mail at kmetzscs@hillwpos.hill.af.mil.

RONALD J. CLAY Ch, Mat Sci & Eng Lab Section Tech & Ind Support Directorate

- 2 Attachments:
- 1. Averages/Charts
- 2. Data



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C1000	<u>Averages</u>	
	%Weight Loss	%Hardness Loss
Coated Water	6.3	5.9
Coated Glycol	5.8	6.3
Coated K-acetate	25.3	18.8
Coated Na-formate	8.6	16.2
Coated No-acetate	9.6	8.6
Coated Urea	7.1	12.5
Non-coated Water	31.5	32.0
Non-coated Glycol	45.4	24.0
Non-coated K-acetate	42.2	15.7
Non-coated Na-formate	<b>e</b> 48.3	18.5
Non-coated Na-acetate	54.6	22.4
Non-coated Urea	37.3	33.2

C2000	<u>Averages</u>	
	%Weight Loss	%Hardness Loss
Coated Water	2.2	6.1
Coated Glycol	1.7	6.5
Coated K-acetate	39.4	24.5
Coated Na-formate	8.8	11.9
Coated Na-acetate	6.5	12.1
Coated Urea	2.6	1.8
Non-coated Water	12.3	24.4
Non-coated Glycol	42.8	36.4
Non-coated K-acetate	36.7	20.7
Non-coated Na-formate	38.8	16.1
Non-coated Na-acetate	51.1	24.5
Non coated Urea	17.0	25.1

C2110	Averages	
	<b>%Weight Loss</b>	%Hardness Loss
Coated Water	1.1	2.2
Coated Glycol	0.8	3.2
Coated K-acetate	16.5	19.5
Coated Na-formate	1.5	6.8
Coated Na-acetate	1.5	11.6
Coated Urea	1.5	1.7
Non-coated Water	25.2	18.9
Non-coated Glycol	43.4	23.5
Non-coated K-acetate	38.3	13.5
Non-coated Na-formate	39.3	8.7
Non-coated Na-acetate	55.8	8.1
Non-coated Urea	34.3	26.8

Attachment I /Deicing ReportAverages and Charts, Page 1

C4000	<u>Averages</u>	
	% Weight Loss	%Hardness Loss
Coated Water	0.6	-0.7
Coated Glycol	0.5	2.1
Coated K-acetate	1.2	0.2
Coated Na-formate	0.4	1.6
Coated Na-acetate	0.4	0.5
Coated Urea	0.8	0.2
Non-coated Water	1.4	3.3
Non-coated Glycol	1.4	3.5
Non-coated K-acetate	7.9	0.9
Non-coated Na-formate	2.2	6.3
Non-coated Na-acetate	3.3	4.7
Non-coated Urea	1.5	5.3

SPK	<u>Average</u>	
	%Weight Loss	%Hardness Loss
Coated Water	1.4	3.3
Coated Glycol	1.4	3.5
Coated K-acetate	7.9	0.9
Coated Na-formate	2.2	6.3
Coated Na-acetate	3.3	4.7
Coated Urea	1.5	5.3
Non-coated Water	15.9	14.4
Non-coated Glycol	56.1	31.1
Non-coated K-acetate	40.2	14.7
Non-coated Na-formate	55.4	17.3
Non-coated Na-acetate	60.3	15.1
Non-coated Urea	17.7	14.2

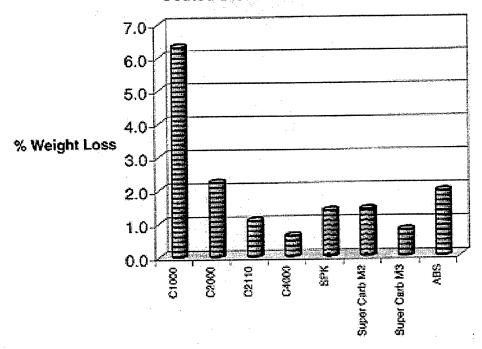
Super Carb M2	<u>Averages</u>	
•	%Weight Loss	%Hardness Loss
Coated Water	1.4	2.9
Coated Glycol	1.6	1.9
Coated K-acetate	8.0	0.0
Coated Na-formate	6.7	3.8
Coated Na-acetate	2.8	1.7
Coated Urea	1.7	2.9
Super Carb		
Non-coated Water	17.8	18.9
Non-coated Glycol	47.1	25.9
Non-coated K-acetate	42.1	18.5
Non-coated Na-formate	46.2	15.8
<b>Non-coated Na-acetate</b>	53.0	14.8
Non-coated Urea	34.0	26.3

Attachment 1 / Deicing Report/Averages and Charts, Page 2

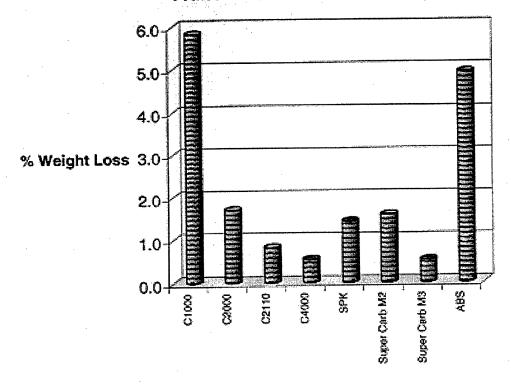
Super Carb M3	Avera	ages
	%Weight Loss	%Hardness Loss
Coated Water	0.8	-0.7
Coated Glycol	0.5	-0.9
Coated K-acetate	0.3	-1.0
<b>Coated Na-formate</b>	0.5	-2.1
Coated Na-acetate	0.5	-0.7
Coated Urea	0.7	-1.6

ABS	<u>Averages</u>					
	%Weight Loss	%Hardness Loss				
Coated Water	1.9	12.4				
Coated Glycol	4.9	13.4				
Coated K-acetate	2.6	18.1				
Coated Na-formate	0.5	0.5				
Coated Na-acetate	0.4	-5.7				
Coated Urea	0.8	-3.4				
Non-coated Water	28.0	53.8				
Non-coated Glycol	50.2	56.8				
Non-coated K-acetate	40.6	40.5				
Non-coated Na-format	<b>e</b> 47.5	26.4				
Non-coated Na-acetate	e 59.4	21.6				
Non-coated Urea	50.6	33.8				

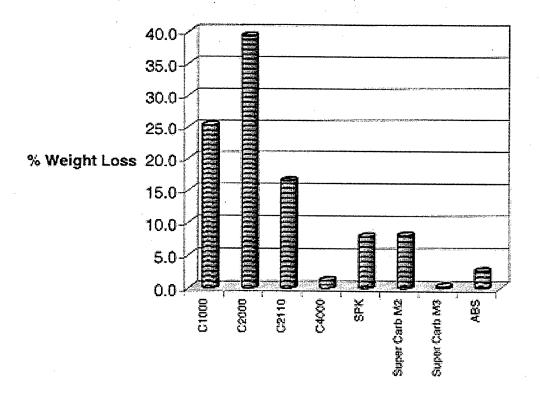
## **Coated Disks Contaminated in Water**



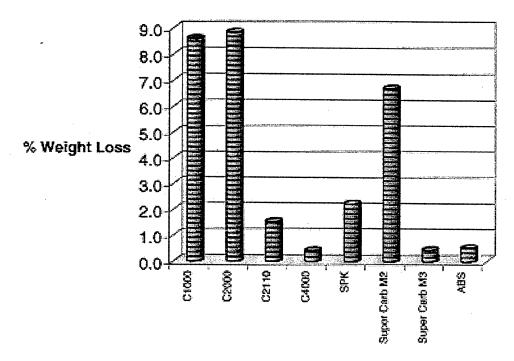
# Coated Disks Contaminated in Glycol Mix



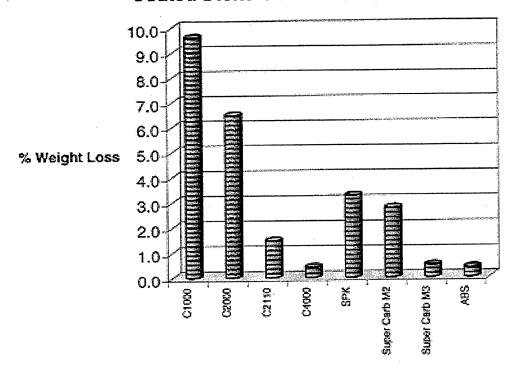
## Coated Disks Contaminated in Potassium Acetate



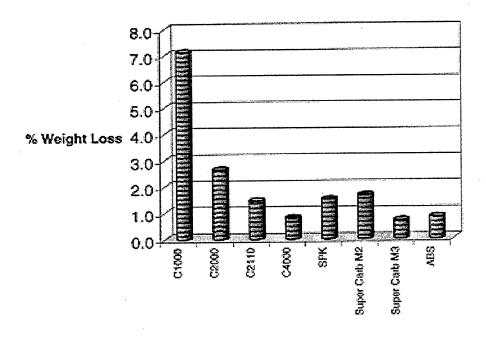
# **Coated Disks Contaminated in Sodium Formate**



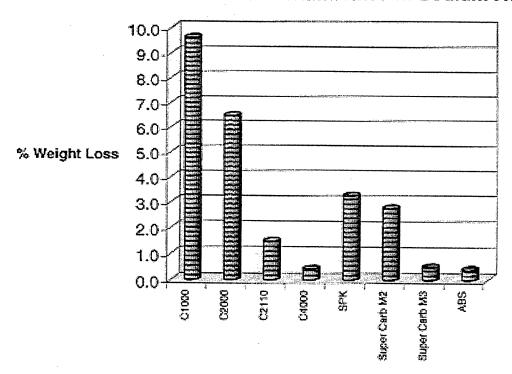
# Coated Disks Contaminated in Sodium Acetate



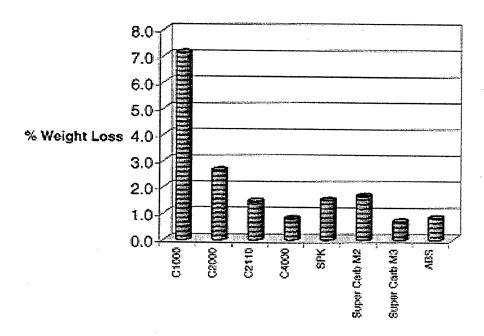
#### Coated Disks Contaminated in Urea



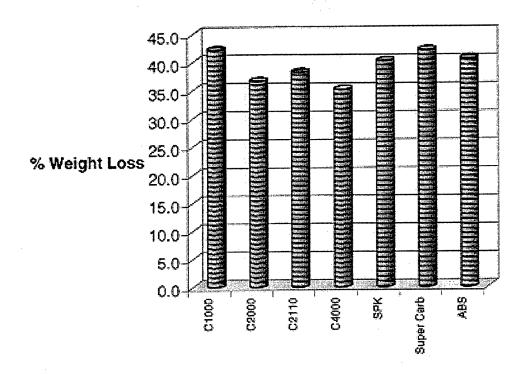
# Coated Disks Contaminated in Sodium Acetate

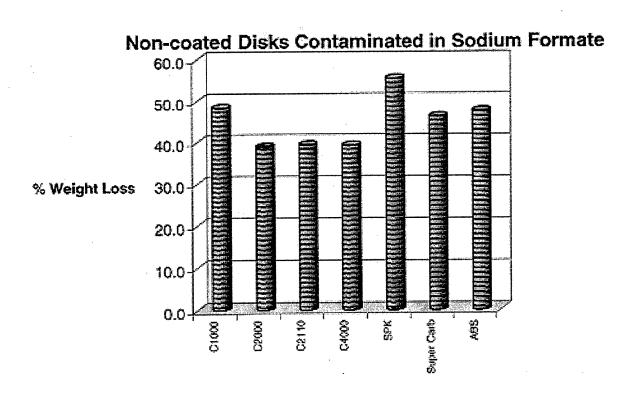


#### Coated Disks Contaminated in Urea



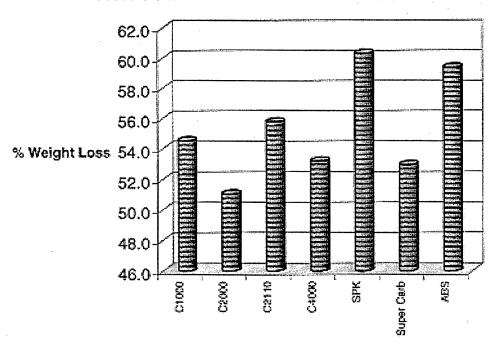
# Non-coated Disks Contaminated in Potassium Acetate



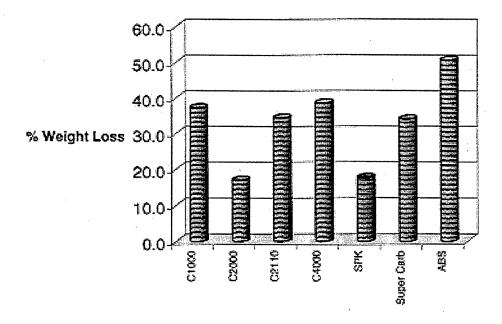


Attachment 1/Deicing Report/Averages and Charts, Page 8

## Non-coated Disks Contaminated in Sodium Acetate



## Non-coated Disks Contaminated in Urea



Attachment 1/Deicing Report/Averages and Charts, Page 9

	station and the same is a second second second second second second second second second second second second			ied C 1000			*** 3
Coupon No.	Delcer water	Initial Weight^ 18.4933	Final Weight*	% Loss 6.5	Initial Hardness≛ 85	Final Hardness*	* Loss 5.0
2	water	183108	17.1858	6.1	85	76	10.6
3	water	18.5179	17.4074	6.0	86	83	3.5
	water	18.5282	17.266	6.8	<b>86</b>	84	23
5	water	18.3518	17.2263	6.1	86	80	7.0
6	glycol mix glycol mix	18.2588 18.5225	17.2074 17.5273	5.8 5.4	85 86	83 81	2.4 5.8
8	glycol mix	18.4757	17.4683	5.5	87	83	4.6
9	glycol mix	18.534	17.5153	5.5	86	82	4.7
10	glycol mix	18,4369	17.1317	7.1	85	73	14.1
11	K-acetate	18.3467	14,6399	20.2	84	82	2.4
12 13	K-acetate K-acetate	18.4223 18.3954	14,518 12,1264	21.2 34.1	88 86	86 60	23 30.2
14	K-acetate	18,3411	13.6481	25.6	86	71	17.4
15	K-acelalo	18,1635	11.3835	37.3	86	50	41.9
16	Na-tormate	18.4987	17.1714	7.2	87	80	8.0
17	Na-formate	18.4092	16.6337	9.6	87	<i>7</i> 5	13.8
18	Na-formate	18.3067	16.6811	9.3	87	65	25.3
19 20	Na-formate Na-formate	18.4707 18.4875	16.9737 16.8677	8.1 8.8	86 85	73 69	15.1 18.8
21	Na-acetate	18.2638	16.4839	9.7	87	82	5.7
22	Na-acetate	18.4855	16.965	8.2	85	84	12
23	Na-acetate	18.2157	16.3921	10.0	87	81	6.9
24	Ne-acetate	18.4372	16.8741	8.5	<b>6</b> 5	74	12.9
25 26	Na-acetate Urea	18.4064 18.413	16.256 17.1451	11.7 6.9	87 89	73 75	16.1 14.8
20 27	Urea	18.4566	17,1685	7.0	86	74	14.0
28	Urea	18.442	17.2091	6.7	86	75	12.8
20	Urea	18.3294	17.1252	6.6	87	80	8.0
30 32#	Urea	18.3522	16.7839	8.5	<b>8</b> 5	74	12.9
33	water water	18.2505 18.1522	8.4622 11.1533	53.6 38.6	<b>85</b> 85	<b>49</b> <b>69</b>	42.4 18.8
34	water	18.3564	13.8078	24.6	86	68	20.9
35	water	18.2315	12.6975	30.4	85	65	27.1
36	water	18.3572	12.4325	32.3	85	89	37.6
37	glycol mix	18.4185	9.504	48.4	<b>8</b> 5 <b>8</b> 5	60	29.4
38 39	glycol mix glycol mix	18.2319 19.3887	9.3472 10.7649	48.7 44.5	<b>8</b> 6	<del>6</del> 0 79	29.4 8.1
40	glycol mix	18.3338	10.9959	40.0	85	75	11.8
41	glycol mix	18.3625	5.4119	70.5	87	65	25.3
42	K-acetate	18.2739	5.1131	72.0	87	76	12.6
43	K-acetate K-acetate	18:3524	10.56	42.5	85 87	75 74	11.8 14.9
44 45	K-acetate	18.4315 18.4546	10.6236 10.7309	42,4 41.9	67 86	66 66	23.3
46	K-acetate	18.3952	10.6112	42.3	86	60	30.2
47	Na-formate	18.4456	10.05	45.5	86	72	16.3
48	Na-formate	18.4014	9.6536	47.5	87	70	19.5
49	Na-formate Na-formate	18.2243	8.9737	47.0	86 <b>8</b> 5	79	8.1 17.6
50 51	Na-formate	18.0948 18.3632	8,4798 4.6347	53.1 74.8	87	70 69	20.7
52	Na-acetate	17.9844	4,347	75.8	85	66	22.4
53	Navacetate	18,3309	7.7467	57.7	84	damaged surface	N/A
54	Na-acetate	19.3116	8.7201	52.4	85	damaged surface	N/A
55 50	Na-acetale	18.3379	9.0109	50.9 57.0	88 97	damaged surface	N/A
56 57	Ma-acetate Urea	18.2969 18.4563	7.8251 12.3166	57.2 33.3	<b>87</b> 88	damaged surface 55	N/A 37.5
58	Urea	18.1828	11.958	34.2	85	58	31.8
59	Urea	18.4718	11.0989	40.0	87	74	14.9
60	Urea	18.3445	10.7069	41.5	86	76	11.6
61	Urea	18.2846	8.5177	53.4	86	60	30.2
*Coate	od 1-30	#Non-cos	sted 32-61	^Weigh	t in Grams (g)/Hardnes	as is Shore D	

			Allied	C 2000			
Coupon No.	Deicer	Initial Weight^	Final Weight^	% Loss	Initial Hardness*	Final Hardness*	% Loss
	water	19.1877	18.7466	2.3	83	79	4.8
2	water	19.818	19.6627	0.7	81	<b>74</b>	8.6
3	water	19.979	19.564	2.0	81	80	12
	water	19.7815	19:303	2.4	79	76	3.8
5	water	20.0526	19.6285	2.1	83	77	7.2
6	glycol mix	19.793	19.4856	1.6	81	72	11.1
7	glycol mix	20.1178	19.7696	1.6	81	76	6-2
8	glycol mix	19,6342	19.2949	1.7	82	79	3.7
9	glycoll mix	19.6807	19.3403	1.7	79	79	0.0
10	glycol mix	19.7729	19.4085	1.6	88	74	<del>6.8</del>
11	K-acetate	19.4231	9.3997	51.6	73	damaged surface	NVA
<b>j</b> 12	K-acetate	19.052	9.6279	49,5	75	50	33.3
13	K-acetate	19.8428	14,0817	29.0	80	68	15.0
14	K-acetate	19.8384	11.9736	39.6	83	62	25.3
15	K-acetate	19,4403	12,6418	35.0	76	damaged surface	NVA
16	Na-formate	19.759	18.4405	8.7	80	70	12.5
17	Na-formate	18.5747	17.6776	4.8	81	76	6.2
18	Na-formate	19.7037	19.084	9.1	82	68	17.1
19	Na-formate	19.6764	17.4408	12.3	78	73	6.4
20	Na-formate	20.4737	18.0965	11.6	80	66	17.5
21	Ne-acetate	19.9781	19.0323	4.7	80	78	2.5
22	Na-acetate	20.0916	19.2035	4.4	82	72	12.2
23	Na-acetate	19.651	17.9888	8.5	84	75	10.7
24	Na acetate	20.5838	16.7634	18.5	84	70	16.7
25	Na-acetate	20,0366	18.3786	8.3	81	74	8.6
26	Unca	20.0866	19.6842	2.0	82	81	1.2
27	Urea	19.617	19.123	2.5	83	79	4.8
28	Urea	20.3069	19.8001	2.5	81	80	1.2
29	Urea	19.8832	19.3102	2.9	78	78	0.0
30	Urea	19.6036	18.9518	3.3	<b>8</b> 5	72	15.3
32#	water	19.8397	17.1226	13.7	82		28.0
33	weter	19.7929	17,4914	11.6	82	60	<b>26.8</b>
34	water	19.4486	17.1788	11.7	82	68	19.5
35	water	19.6428	17.2083	12.4	82	es ·	23.2
36	water	19.579	17.211	12.1	80	74	7.5
37	gilyool mix	19.8634	10.8739	45.3		59	25.3
38	glyoci mix	19.9217	11.0066	44.8	80	52	35.0
39	glycol mix	19.6496	13.019	33.7	82	47	42.7
40	glycol mix	19.5784	10.2997	47.A	<b>8</b> 0	46	42.5
41	glycot mix	18.973	7.9861	61.2	80	40	50.0
42	K-acetate	19.5231	4.8104	75.4	<b>83</b>	62	25.3
43	K-acetate	19.6782	10.9176	44.5	82	68	17.1
44	K-acetate	19.4402	12_4963	35.7	<b>6</b> 3	68	18.1
45	K-acetate	20.3128	13,6507	32.8	. 83	74	10.8
46	K-acetate	19,7709	13.122	33.6	81	63	22.2
47	Na-formate	19.8766	11.8357	40.5	79	70	11.4
48	Na-formate	19.5446	12.5619	35.7	80	73	8.8
49	Na-formate	19.9017	12.5383	37.0	79	77	2.5
50	Na-formate	20.2235	11.6933	42.2	81	65	19.8
51	Na-formate	19.7754	7.927	59.9	81	<b>6</b> 1	24.7
52	Ne-acetate	19.7168	5.019	74.5	83	74	10.8
53	Na-acetate	19.2603	9.3936	51.3	63	50	39.8
54	Na-acetate	20.0235	10.2024	49.0	82	71	13.4
55	Na-acetate	19.5879	11,203	42.8	83	60	27.7
56	Na acetate	19,6614	7.6516	61.1	84	58	31.0
57	Urea	19.6931	16.6181	15.6	83	<b>6</b> 8	20.5
58	Urea	19.9073	16.3746	17.7	83	65	21.7
59	Urea	19.8846	17.1842	13.6	81	61	24.7
60	Urea	19.9319	16.6636	16.4	81	60	25.9
	LX82	20.3698	16.0035	21.4	82	55	32.9
*Coss	d 1-30	#Non-cost			ht in Grams (g)/Hardn		Na Main e Mai
2444 to 1000 t		the above Algorith			here on a resolution of Miles and property to	is careie v	*

oupon No.	Deicer	Initial Weight^	<u>Allie</u> Final Weight*	% Loss	Initial Hardnes	TAXABLE PROPERTY OF THE PERSON NAMED AND POST	**********************
1.	water	20.5182	20.3319	0.9	81	80	12
2	water	20.4396	20.2457	0.9	83	79	4.8
3	water	20.3326	20.1204	1.0	80	90	0.0
4	water	20.6243	20.4221	1,0	. 62	<b>.</b>	4.9
5	water	20.2583	19.9662		80	<b>50</b>	0.0 6.3
6	glycol mix	20.3496	20.1576	0.9	79	74 81	0.0
7	glycol mix	20.4176	20.2562	8.0	<b>8</b> 1 83	79	4.8
8	glycol mix	20.4168	20.2728	0.7 0.8	ಕು 83	82	1.2
9	glycol mix	20.3747	20.2218 20.246	0.0	&5 81	76	3.7
10	glycol mix	20.431	16.4333	0.2 19.8	83	82	1.2
11	K-acetate K-acetate	20,4959 20,3846	17.3831	14.7	82	50	39.0
12	K-acetate	20,7525	15.9581	23.1	84	48	42.9
13	K-acetate	20,1929	17.869G	11.9	85	82	3.5
14 15	K-acetate	20.5377	17.8292	13.2	83	74	10.6
16	Na-iomate	20.4078	19.8371	2.8	<b>8</b> 0	73	8.8
17	Na formate	20.8079	20.5286	1.3	80	77	3.8
18	Na-formate	20.4336	20.1566	1.4	79	65	17,7
19	Na-formate	20.5978	20.387	1.0	80	90	0.0
20	Na-formate	20.4624	20.2073	1.2	82	79	3.7
21	Na-acetate	20.4377	20.2577	0.9	80	75	6.3
22	Na-acetate	20.6391	20.3415	1.4	82	67	18.3
23	Na acetate	20.6871	20.4343	1.2	82	69 	15.9
24	Na-acetate	20.4929	19.8966	2.9	79	71	10.1
25	Ne-acetate	20.3586	20.1668	0.9	83	77	7.2 8.5
26	Urea	20.4256	20.1319	1.4	82	75 78	0.0
27	Urea	20.2871	20.0196	1.3	78 80	60	0.0
28	Urea	20.3659	20.0505	1.5 1.6	79	79	0.0
29	Urea	20.3136	19.9918 20.1222	1.4	80	80	0.0
30	Urea.	20.4051 20.2592	13.5208	**************************************		6	19.8
32e	water water	20.292 20.103	14.2288	29.2	79	52	34.2
33 34	water	20.0296	16.866	15.8	81	66	18.5
35 35	water	20.1273	15.052	25.2	81	70	13.6
36	water	20.2081	15.6851	22.4	82	75	8.5
37	glycol mix	20.0688	11.219	44,2	80	63	21.3
38	głycel mix	20.1521	12:2185	39.4	. 81	65	19.8
39	glycol mix	19.8584	12.0461	39.3	82	65	20.7
40	glycol mix	20.3705	10.0463	50.7	83	60	26.8
41	glycol mix	20.4574	6.0964	70.2	83	59	28.9
42	K-acetate	20.2543	5.7272	71,7	83	70	15.7 13.4
43	K-acetate	20.4065	11,2355	44.9	82	71 69	13.8
44	K-acetate	20.3597	12.878	36.7	80 82	72	12.2
45	K-acetate	20.2486	12,8501	36.5 34.8	80	70	12.5
46	K-acetale	20.2033	13.1719	36.4	82	76	7.3
47	Ne-formate	20.2642	12.8886 12.573	37.3	79	75	5.1
48	Na-formate Na-formate	20.0599 20.229	11.6074	37.8	79	69	12.7
49 50	Na-formate	20.0884	10.8824	45.8	81	72	11.1
50 51	Na-lormate	20.0221	5.0665	74.7	81	75	7.4
52	Na-acetate	20.2208	4.2747	78.9	80	75	5.3
53	Na-acetale	20,2519	9.15	54.8	80	75	6.3
54	Na-acetate	20,2082	9.4312	53.3	79	76	3.8
55	Na-acetate	20.0209	8,5601	57.2	82	82	24.4
 56	Na-acetate	20.0611	8,4877	57.7	81	68	16.0
57	Urea	20.2619	13.9787	31.0	81	61	24.7
58	Urea	19.7342	11,8682	39.9	81	70	13.6
59	Urea	20.0907	12.9971	35.3	82	60	26.8
60	Urea	20.2125	16.586	17.9	83	57	31.3
	and the second second	20.1793	10.5771	47.6	80	.50	37.5

Initial Weight ^ Final Weight\* Coupon No Delcer % Loss Initial Hardness^ Final Hardness^ % Loss 20.4873 20.3551 0.5 84 85 water -12 2 20.668 20.59 84 0.0 water 0.5 84 3 water 20.017 19.8784 0.7 83 84 -12 4 water 20.3803 25 20.2482 0.6 85 0.0 5 20.0674 19.9414 water 0.6 84 85 -1.2 6 20,1162 20.007 0.5 83 glycol mix 84 -1.2 7 glycol mix 20,4203 20.2862 0.7 86 84 23 a 20.3567 20.2576 87 glycol mix 0.5 83 4.6 ğ 19.98 19.8637 86 1.2 glycol mix 0.6 85 10 19.9642 19,8774 0.4 85 3.5 glycol mix 82 K-acetate 19.9542 0.1 11 19.9365 86 85 0.0 12 K-acetate 19,4708 18,9996 24 84 84 0.0 13 K-acetate 20.7161 20.6924 0.1 87 88 1.1 14 K-scetate 19.8327 19.8196 0.1 87 86 1.1 15 K-acetate 18.9019 18.2932 3.2 85 86 -1.2 16 Na-formate 19.793 19.7185 0.4 88 85 3.4 17 Na-formate 20.4221 20.3189 0.5 85 85 0.0 Na-formate 18 20.3502 20.2546 86 0.5 85 1.2 13 Na-formate 20.0509 19.9803 0.3 83 83 0.0 20 Na-formate 19.7495 19.6672 0.4 86 83 3.5 Na-acetate 21 20.4781 85 20.4073 0.3 85 0.0 22 Na-acetate 19.869 19,7643 0.5 84 84 Q.O 23 Na-acetate 20.3725 20.206 0.3 85 85 0.0 24 Na acetate 20.5624 20.6261 0.3 86 86 0.0 25 Na-acetate 20.0454 19.9235 85 0.6 83 2.4 26 Urea 20.1342 20.0081 84 83 0.6 1.2 27 Ures 20,7591 20.6128 0.7 85 63 2.4 28 Urea 20.8617 20.7156 0.7 85 86 -1.2 29 Urca 19.7309 19,4999 1,2 83 **B4** 12 30 Urea 20,1114 19.9564 0.8 83 83 0.0 32# water 19.7134 12.8485 34.8 82 77 5.1 33 water 19.9846 11.2279 43.8 85 77 9.4 34 20.3282 77 water 12.2318 39.8 86 10.5 35 water 20.245 12.3839 38.8 76 86 11.6 36 20.0145 water 13.1282 34.4 86 75 12.8 37 glycol mix 19.9757 12.3110 38.4 88 81 9.0 38 glycol mix 19.4971 11,3721 41.7 85 74 12.9 39 19.6699 glycol mix 10.371 47.3 83 72 13.3 40 glycol mix 19.6304 52.0 9,4303 84 68 19.0 41 19.8319 glycol mix 3.8526 80.6 85 68 20.0 42 K-acetate 19.3295 5.6658 70.7 88 76 **13.6** 43 K-acetate 20,4143 12.8641 37.0 86 78 9.3 44 K-acetate 20.2798 12.9337 36.2 84 68 19.0 45 K-acetate 19.7948 13,414 32.2 85 71 16.5 46 K-acetate 19.5263 12.6534 35.2 89 76 14.6 47 Na-formate 20.0661 11.8993 40.7 88 72 16.3 48 Na-formate 19,4648 12.667 34.9 87 76 12.6 Na-formate 49 19.6497 11.9776 35.5 85 76 10.6 50 Na-formate 86 77 19.9547 10.867 45.5 10.5 51 Na-formate 19,893 4.5968 76.9 66 76 11.6 52 Na-acetale 19.8012 2.9475 85.1 87 60 31.0 53 Na acetate 20.1271 9.3947 53.3 85 75 11.8 54 Na-acetate 19,7097 9.2105 53.3 89 74 16.9 55 Na-acetate 19.4365 9.157 52.9 **8**9 75 15,7 56 Na acclaic 9.4502 20.242 53.3 78 88 136 57 Urea 19.9593 10.6496 46.6 89 74 16.9 59 Lives 11.0999 42.9 19.4556 83 74 10.8 59 **Uven** 19,7094 14,412 26.9 84 71 15.5 60 Urea 19.26 11.9322 83 38.0 74 10.8 61 Urea 20.0375 3.3994 83.0 83 65 21,7 Coaled 1-30 #Non-coated 32-61 "Weight in Grams (g)/Hardness is Shore D

Allied C 4000

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Coupon No.	<u>Deicer</u>	Initial Weight*	Final Weight^	% Loss	Initial Hardness*	THE RESERVE AND ADDRESS OF THE PERSON NAMED IN COLUMN TWO	
K2-1*	water	19.0829	18.8428	1.3	86	80	7.0
102-2	water	19.0071	18.7802	1.2	<b>96</b>	82	47
К2-3	water	18.8023	18.5297	1.4	85	84	12
K2-4	water	19.1204	18.8616	1.4	883	<b>82</b>	0.0
K2-5	water	18.8526	18.5638	1.5	26	83	3.5
K2-6	glycol mix	18,7744	18.5175	1.4	86	82	4.7
K2-7	glycol mix	18.8343	18.5729	1,4	86	<b>8</b> 3	3.5
K2-8	glycol mix	19.1718	18.9064	1.4	85	82	3.5
K2-9	glycol mix	18.8271	18.5415	1.5	84	84	0.0
K2-10	glycol mix	19.3036	19.0244	1.4	88	83	5.7
K2-11	K-acetate	19,2658	18.2838	5.1	82	82	0.0
K2 12	K-acetate	19.0086	18.5154	2.6	79	79	0.0
K2-13	K-acetate	19.1296	15.8191	17.3	86	86	0.0
K2:14	K-acetate	18.9164	17.7032	6.4	86	83	3.5
K2-15	K-acetate	19.0847	17.5744	7.9	88	87	1.1
K2-16	Na-formate	19,2308	18.7671	2,4	<b>65</b>	81	4.7
K2-17	Na-formate	18.7612	18.3369	2.3	87	81	6.9
K2-18	Na-formate	18.8029	18.3057	2.6	<b>8</b> 6	79	8.1
K2-19	Na-formate	18.7215	18.3526	2.0	86	76	9.3
K5-50	Na-formate	18.9267	18.5724	1,9	85	83	2.4
K2-21	Na ocetate	18.8334	18.525	1.6	85	77	9.4
K2-22	Na-acetate	18.5137	17.4452	5.8	86	82	4.7
K2-23	Na-acetate	18,704	18.0394	3.6	83	78	6.0
K2-24	Na acetate	18.677	18.1594	2.8	84	-81	3.6
K2-25	No acetale	18.6823	18.2	2.6	81	81	0.0
K2-26	Urea	18.8003	19.4848	1.7	86	.80	7.0
K2-27	Urga	18.8626	18.5768	1.5	88	<b>82</b>	6.8
K2-28	Urea	19.1429	18.8656	1.3	86	81	5.8
K2-29	Urea	18.9139	18.6611	1.3	83	82	1.2
K2-30	Urea	18,8196	18.5143	1.6	87	82	5.7
K1-1#	water	18.8954	15.8285	16.2	89	<b>75</b>	15.7
Ki-2	water	18.5062	17.3715	6.1	84	82	2.4
K1-3	water	18.9666	15.4043	18.8	85	65	23.5
K1-4	water	18.3718	15.7409	14.3	26	74	14.0
K1-5	water	18.9632	14.3545	24.3	85	71	165
K1-6	glycol mix	18.3238	7.8657	57.1	89	53	36.1
K1-7	glycol mix	18.757	8.312	55.7	82	82	0.0
K1-8	glycol mix	18.8762	8.3859	55.6	84	47	44.0
K1-9	glycol mix	18.8076	8.8802	52.8	87	61	29.9
K1-10	glycol mix	18.3989	7,4511	59.5	79	43	45.6
K1-11	K-acetate	18.7538	4.698	75.0	86	71	17.4
K1-12	K-acetate	19.0904	10,5298	44.8	87	72	17.2
K1-13	K-acetate	18.5096	11.158	39.7	83	75	9.6
K1-14	K-acetate	18.7197	11,2975	39.6	74	70	5,4
Ki-15	K-acetate	19.0249	12,0833	36.5	88	67	23.9
K1-16	Na-formate	19.0695	9.6601	49.3	86	73	15.1
K1-17	Na-formate	18.2818	8.9924	50.8	86	76	11.6
K1-18	Na-formate	18.5141	8.4129	54.6	82	64	22.0
K1-19	Na-formate	18.6977	6.1983	66.8	96	72	16.3
K1-20	Na-formate	18.708	1.1156	94.0	84	66	21.4
K1-21	Na-acetate	18.5294	3.1285	83.1	81	73	9.9
K1-22	Ne ecetate	18,4319	8.3951	54,5	83	66	20.5
K1-22	Navacetate	18,4907	7:7832	57.9	86	78	9.3
K1-24	Ne-acetate	18.7291	7.3196	60.9	87	73	16.1
K1-25	Na-acetate	18.652	5,9745	68.0	87	70	19.5
K1-26	Urea	18.5418	14.6213	21.1	86	68	20.9
K1-20	Urea	18.7726	18.1723	3.2	86	76	11.6
K1-27	Urea	19.0317	16.0011	15.9	87	70	19.5
K1-20	Urea	18.6046	14.6072	21.5	82	73	11.0
K1-29 K1-30	Urea	18.4163	16.1458	12.3	86	79	8.1
Maniministi ( 4 ) i 4 ) mariemange a 4 a	,_,_, <del>,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,</del>	44	ated K1:1-30		ight in Grams (g)/Ha		
Coste	dK2:1-30	#IAODACO	uppi i i i i jili	ं विश	Server and Antonion of Mile , soil	The second section of the second section is a second section of the second section sec	

Coupon No. initial Weight^ Final Weight 1/2 Loss Initial Hardness\* Final Hardness\* Deicer % Loss 18,7046 18,4707 1.3 62 80 2.4 C2-1 water 1.2 82 81 1.2 C5-5 water 18.9303 18.6968 4,7 **B**5 81 C2-3 water 18,6536 18.3628 1.6 C2-4 water 18.6617 18.3669 84 81 3.6 1.5 C2-5 water 19.0097 18,7543 1.3 84 82 2.4 C2-6 18,7192 18.2984 22 81 80 1.2 glycol mix C2-7 19.015 1.2 84 84 0.0 glycol mix 18.7626 C2-8 glycol mix 18,6698 18.3836 1.5 84 80 4,8 C2-9 glycol mix 18.8446 18.6007 1.3 83 82 1.2 C2-10 glycol mix 18.5934 18294 1.6 85 83 2.4 C2-11 K-acetate 18,6393 17.2479 7.5 85 65 0.0 C2-12 K-acetate 18.4843 17.3587 6.1 81 81 0.0 C2-13 K-acelate 18,3152 14.6044 20.3 85 85 0.0 C2-14 K-acetate 19.5057 18.1078 22 85 **B**\$5 0.0 C2-15 K-acetate 18.87 18.0743 4.2 81 81 0.0 C2-16 Na-formate 18.7137 17.3028 7.5 82 75 8.5 C2-17 Na-formate 18.5734 15.7157 15.4 85 82 3.5 C2-18 Na-formate 28 85 81 4.7 18.8468 18.3565 C2-19 Na-formate 18.7344 17.6593 5.7 84 82 2.4 CS-50 Na-formate 19.1033 18,5662 23 80 80 0.0 Na-acetate 3.6 81 (22-21 18.8629 16.1805 81 0.0 C2-22 Na-acetate 18.7965 2,7 85 82 3.5 18.2868 0.0 C2-23 Na-acetate 18,7065 17.9417 4.1 82 82 C2-24 Na-acetate 18.9326 1.5 85 82 35 18.6438 C2-25 Na-acetate 83 18.9636 18.6162 1.9 82 C2-26 19,1154 18.7924 85 83 24 Urea 1.7 C2-27 85 79 Urea 19,0007 18.6976 1.6 7.1 C2-28 Urea 18,6886 18.3945 1.6 80 79 1.3 C2-29 19\_027B 18.6968 63 81 Urea 1.7 2.4 C2-30 18,3068 80 Urea 18.615 1.6 79 1.3 C1-1# 62 water 18.6159 13.5403 27.3 84 26.2 Ç1-2 water 18.3282 15,6500 14.6 82 68 17.1 C1-3 water 18.2481 15.9177 128 80 70 12.5 C1-4 water 18.363 14.4838 21.1 86 65 24.4 C1-5 water 18.2795 15.8336 13.4 84 72 14.3 C1-6 glycol mix 18.1806 10.0649 44.6 79 30 51.9 C1-7 glycol mix 18.4948 9,9762 46.1 81 61 24.7 C1-8 glycol mix 18.2446 9.7962 46.3 83 64 22.9 C1-9 glycol mix 18.3878 8.9169 515 83 63 24.1 C1-10 glycol mix 18.022 85 58 5.076 71.B 31.8 C1-11 K-acetate 83 33.4 62 25.3 18:2532 12.162 C1-12 K-acelale 18.1373 12.0155 33.8 82 22.0 64 C1-13 K-acetate 18,3154 9.6342 82 62 24.4 47.4 K-acetate C1-14 18.364 8.4726 53.9 85 73 14.1 C1-15 K-acetate 18.1415 3.8001 79.1 86 7.0 80 C1-16 Na-formate 18.3673 10.6151 42.2 85 76 10.8 C1-17 Na-formate 85 18.5329 11,238 39,4 79 7.1 C1-18 Na-formate 18.2475 9.953 45.5 81 67 17.3 C1-19 Na-formate 18.4418 7.8116 57.6 85 61 28.2 C1-20 Na-formate 18,6339 2,701 85.5 20 43 46.3 C1-21 Na-acetate 18.5445 82.9 80 61 3.1783 23.8 C1-22 Na-acetale 18.1189 8.9595 50.6 80 72 10.0 C1-23 Na-acolale 18.5366 9.314 49.8 80 72 10.0 C1-24 Na-acetate 18.2285 8.3843 85 74 54.0 12.9 C1-25 Na-acetale 18.1621 7.7137 57.5 86 71 17,4 C1-25 Urea 18.4222 9.3838 49.1 81 44 45,7 C1-27 Urea 18.1318 11.7653 35.1 87 59 32.2 C1-28 Urea 11,7281 35.4 83 63 18.1507 24.1 C1-29 Urea 18.2952 11.8515 36.3 53 63 24.1 C1-30 29.4 Urea 18.5177 13.0764 84 83 25.0 \*Coated C2:1-30 #Non-coated C1:1-30 "Weight in Grams (g)/Hardness is Shore D

**BFG Super Carb M2** 

BFG Super Carb M3									
Coupon No.	Deicer	Initial Weight*	Final Weight*	% Loss	Initial Hardness^	Final Hardness*	% Loss		
C3-1*	water	20.0221	19,8793	0.7	89	<b>80</b>	-1.1		
C3-8	water	19.9842	19.8062	0.8	<b>. 69</b>	80	0.0		
C3-3	water	19,6797	19.556	0.6	89	80	-1.1		
C3-4	water	19,7913	19.6346	0.8	68	90	-2.3		
C3-5	water	19.6128	19.436	0.9	90	<b>89</b>	(1.1)		
C3-6	glycol mix	19.7162	19.6016	0.6	90	89	1.1		
C3-7	glycol mix	19.3463	19. <b>2</b> 573	0.5	86	90	-4,7		
C3-8	glycol mix	19.7819	19.6696	0.6	.89	89	0.0		
C3-9	glycol mix	19.8943	19.7923	0.5	90	91	-1.1		
C3-10	glycol mix	19.7172	19.6199	0.5	90	90	0.0		
C3-11	K-acelate	19,5474	19,5001	0.2	69	91	-2.2		
C3-12	K-acetate	19.761	19.744	0.1	89	90	-1,1		
C3-13	K-acetate	19,7708	19,689	0.4	87	89	-2.3		
C3-14	K-acetate	20.0282	19.98	0.3	91	86	5.5		
C3-15	K-acetate	19.9176	19.8788	0.2	85	89	-4.7		
C3-16	Na-formate	19.6713	19.59	0.4	\$8	90	-2.3		
C3-17	Na-formate	19.9948	19.9222	0.4	88	90	-2.3		
C3-18	Na-formate	19,6039	19.5101	0.2	87	91	-4.6		
C3-19	Na-formate	19.616	19.5201	0.8	91	89	2.2		
C3-20	Na-formate	19.5299	19.4299	0.5	<b>85</b>	88	-3.5		
C3-21	Na-acetate	19,7188	19.6273	0.5	91	91	0.0		
C3-22	Na-acetate	19.6933	19.5806	0.6	87	- 86	-1.1		
C3-23	Ma-acetate	19,6684	19,551	0.6	89	89	0.0		
C3-24	Ne-ecetate	19.3881	19,3095	0.4	87	89	-2.3		
C3-25	Na-acatate	19,0075	18,922	0,4	89	89	0.0		
C3-26	Urea	19,4402	19.2938	0.8	87	90	-3.4		
C3-27	Urea	19.3546	19.2364	0.6	88	90	-2.3		
C3-28	Uree.	19.542	19.4263	0.6	87	90	-3.4		
C3-29	Urea	19.6806	19.5062	0.9	89	89	0.0		
C3-30	Urea	19.317	19.1901	0.7	91	90	1.1		

oupon No.	Deicer	Initial Weight^	Final Weight	ABS % Loss	initial Hardness*	Final Hardness^	% Loss
2"	wale	22,6106	22.287	1.4	81	<b>89</b>	-2.5
3	water	22.5202	21,7148	9.6	84	73	13.1
	water	22,9164	22.3073	0.0	<b>85</b>		10,8
5	water	22.489	21.692	2.7			13.3
6 7	ghycol mix	23.4067	22,0398	5.8 = 2	<b>82</b>	78	4.9 17.0
8	glycol mix glycol mix	23.1641 23.0502	21,9275 22,0426	5.3 4,4	84 82	69 78	17.9 4.9
8	glycol mix	22.8839	21.8716	5.3	84	64	23,8
10	glycol mix	23.0002	22.1397	3.7	94	71	15.5
11	K-acetate	22,0388	21.5118	2.4	85	77	9,4
12	K-acetate	22.4595	21.9995	2.0	- 83	74	10.8
13	K-acetate	22,4515	22.2 <del>5</del> 65	0.9	84	60	28.6
14	K-acetate	22.2376	21.0203	5.5	83	.69	16,9
15	K-acetate	22.2435	21.7103	2.4	94	63	25.0
16	Na-formate	23.2214	22.9845	1.0	84	83	1.2
17	Na-formate	23.065	22.8574	1.0	63	83	0.0
18	Na-formate	23.4347	23.3827	0.2	84	82	2.1
19	Na-formate	23.1264	23.054	0.3	84	83	1.2
20	Na-formate	23.0357	23.0186	0.1	<b>e</b> 1	83	-2.5
21	Na-acetate	23,4246	23.3257	0.4	81	36	-6.2
22	Na-acetate	23.282	23,2128	0.3	61	88	-8.6
23	Na-acelate	23.0731	22.6355	1.0	82	85	-3.7
24	Na-acelate	23.0793	23.0734	0.0	- 81	85	-4.9
25	Ne-acetate	23.3858	23,3353	0.2	80	84	-5.0
26	Urea	23.3052	22.9131	1.7	81	87	-7.4
27	Urea	23.985	23.6865	1.2	<b>84</b>	85	-1.2
28	Urea	24.1901	23,9872	0.6	81	85	4.9
29	Urea	23.7568	23.7268	0.1	<b>63</b>	84	-1.2
30	Lkea	24.2083	24,0889	0.5	83	25	2.4
31#	water	22.7106	15.6859	30.9	<b>81</b>		71.6
32	waier	23.263	15.8508	31.9	×	<b>31</b>	62.2
33	water	23.2011	16.3052	29.7	<b>B3</b>	40	51.8
34	water	22.6779	17.3112	23.7	82		56.1
35	water	22.7 23.2435	17.3462	23.6	<b>82</b>	45	45.1
36 37	glycol mix glycol mix	22.4949	9.5846 12.553	57.0	<b>85</b>		77.6
38	glycol mix	22.4537	12.7402	44,2 43.3	91 83	25 38	69.1 54.2
30	glycol mix	22.7183	12.7245	44.0	84		54.8
40	glycol mix	22,4643	8,3989	62.6	88	43	48.2
41	K-acetate	22.416	6.7324	70.0	82	50	39.0
42	K-acetate	23.0601	12.0982	47.5	82	48	41.5
43	K-acetate	22,3544	13.2535	40.7	84	48	42.9
44	K-acetate	23.0486	14.1329	38.7	80	449	40.0
45	K-acetate	22.7545	14,6914	35.4	80	50	37.5
46	Na-formate	22,6123	12.3678	45.3	83	64	22.9
47	Na-iomate	22.7864	12.9106	43.3	83	63	24.1
48	Na-formate	22.3897	11.0485	42.3	81	63	22.2
49	Na-formate	22.6618	<b>9.27</b> 3	59.1	83	56	32.5
50	Na-formate	22.5891	3.2489	85.6	83	58	30.1
51	Na-acetate	22,4878	2.7874	87.6	84	57	32,1
52	Na-acetate	22.7156	8.4378	62.9	80	69	- 13.8
53	Na-acetate	22.3207	9,4848	57.5	82	66	19.5
54	Na-acetate	22.6346	9.6649	57.3	81	64	21.0
55	Na-acetate	22.6533	9,0947	59.9	82	<b>4</b> 3	47.6
56	Urea	22,4606	11,4591	49.0	79	50	36.7
57 50	Urea	22.4503	10.9499	51.2	80	42	47.5
58	Urea	22.5348	11.1153	50.7	83	57	31.3
59	Urea	22.3848	10.832	51.6	79	60	24.1
60	Urea	22.6438	7.3446	67.6	81	57	29.6